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TABLE OF CONTENTS

Vol. 23 August, 1940 No. 8

THE JOURNAL

Effect of Particle Size and Index of Refraction on Reflectance 221
By B. W. King, Jr.

Reactions Taking Place During Smelting of Superopaque Antimony Enamel 225
By B. W. King, Jr., and A. I. Andrews

Development of Opacity in Superopaque Enamels 228
By B. W. King, Jr., and A. I. Andrews

Viscous Properties of Soda-Boric Oxide-Silica Glasses at Temperatures from 900° to 1500°F 232
By B. W. King, Jr., and A. I. Andrews

Short Methods for Chemical Analysis of Glass 235
By P. A. Webster and A. K. Lyle

Investigation of Effect of Heat on Clay Minerals, Illite and Montmorillonite 242
By R. E. Grim and W. F. Bradley

CERAMIC ABSTRACTS

19—8

Abrasives 179
Art and Archeology 180
Cements 182
Enamel 183
Glass 185
Structural Clay Products 189
Refractories 190
Terra Cotta 192
Whiteware 193
Equipment and Apparatus 194
Kilns, Furnaces, Fuels, and Combustion 197
Geology 198
Chemistry and Physics 200
General 204

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EFFECT OF PARTICLE SIZE AND INDEX OF REFRACTION ON REFLECTANCE*

By Burnham W. King, Jr.

Abstract

The effect of particle size on the reflectance of thin semitransparent layers, such as enamels, was studied by using mixtures containing opacifying particles of definite sizes in an oil of known index of refraction. The mixtures were applied on a black glass plate, and the reflectance was measured. The coefficient of scatter per unit surface of the particles was calculated and was found to decrease slowly as the particle size decreased. On the other hand, for the range of sizes studied, the reflectance of the semitransparent layer increased rapidly as the particle size decreased.

I. Introduction

A number of theoretical studies have been made on the scattering of light by small particles. The original work was done by Lord Rayleigh,[1] who studied the scattering of light by particles of extremely small size. Mie[2] calculated the scattering of particles of various sizes, but he was mainly interested in suspensions of colloidal metals. Shoulejkin[3] applied Mie's theory to dielectrics. An extensive theoretical and experimental study of the optical properties of opal glasses was made by Ryde and Cooper,[4] who used Lord Rayleigh's equations as the basis of their theoretical work. Studies have also been made on the effect of particle size on the transparency of very dilute suspensions. Some of the latest investigations were those by Stutz and Pfund[5] and by Stutz.[6] Kubelka and Munk[7] developed equations which have been shown by Judd, Harrison, and Sweo[8] to be useful in expressing the properties of vitreous enamels.

According to theory and according to tests made on thick layers of very dilute suspensions, variations in particle size or in the difference between the refractive index of the suspended material and that of the surrounding medium can cause decided variations in the transparency of a suspension. Such tests, however, may not necessarily be comparable to the results obtained by a reflectance test in which the measurement is made by reflected light. This is because absorption of light by the turbid medium causes low transparency and low reflectancy, whereas a high degree of scattering by the turbid medium causes low transparency and high reflectance. Some difference might also be expected between the results obtained with an opaque enamel, which is a thin layer containing a high concentration of opacifying material, and those obtained by turbidity measurements in which a relatively thick layer of a very dilute suspension is considered.

The object of this investigation was to determine the effect of particle size and index of refraction under conditions approximating those existing in opaque enamels. Some idea would thus be obtained of the practical importance of variations in these factors.

II. Procedure

The first step in this investigation was to obtain some fractions of crystalline material of definite particle size. Milled zircon was selected as the first material to be tried as it is an inert material of high refractive index and the milled product contains a wide range of particle sizes. Zircon of a good grade is nearly colorless and disperses readily in water. The following fractions were obtained with this material by settling in water: 0–1, 1–2, 2–5, 5–10, 10–20, 20–30, and 30–44 microns, 44 microns being the size of the openings in a 325-mesh screen. The particle sizes obtained were calculated from Stokes' law of settling. The curves in Fig. 1 were calculated on this basis.

To obtain a fraction of a given size range, the particles above a certain size were first allowed to settle out. The sample was then decanted, and the suspension was allowed to settle until all those particles above the lower limit had settled out. The liquid was then removed and more water was added. The sample was shaken, and the settling was repeated until the suspension became clear after a definite time of settle. The fractions, examined under the microscope, were found to fall within the calculated limits.

The next step was to mix the particles in some medium having an index of refraction approximating that of glass. For this purpose, glycerine with a refractive index of 1.470 was used, and the mixing was done in an agate mortar or on a glass plate. The mixture was poured on a plate of black glass on which had been placed a $3^1/_2$-inch brass ring. A thin glass plate was pressed on the sample until it was squeezed down

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* Presented at the Forty-First Annual Meeting, American Ceramic Society, Chicago, Ill., April 18, 1939 (Enamel Division). Received March 15, 1940.

[1] Lord Rayleigh, "On the Light from the Sky, Its Polarization and Color," *Phil. Mag.*, **41**, 107, 274 (1871).

[2] Gustav Mie, "Beiträge zur Optik trüber Medien, speziell Kolloidaler Metallösungen," *Ann. Physik*, Series 4, **25** [3] 377–445 (1908).

[3] W. Shoulejkin, "Scattering of Light by Big Colloidal Particles," *Phil. Mag.*, Series 6, **48** [284] 307–20 (1924).

[4] (*a*) J. W. Ryde, "Scattering of Light by Turbid Media: I," *Proc. Roy. Soc.* [London], **A131** [817] 451–64 (1931); (*b*) J. W. Ryde and B. S. Cooper, Part II, *ibid.*, pp. 464–76 (1931); *Ceram. Abs.*, **10** [8] 554 (1931).

[5] G. F. A. Stutz and A. H. Pfund, "Relative Method for Determining Particle Size of Pigments," *Ind. Eng. Chem.*, **19** [1] 51–53 (1927).

[6] G. F. A. Stutz, "Scattering Light by Dielectrics of Small Particle Size," *Jour. Franklin Inst.*, **210** [1] 67–85 (1930); *Ceram. Abs.*, **10** [4] 306 (1931).

[7] P. Kubelka and F. Munk, "Ein Beitrag zur Optik der Farbenstriche," *Z. tech. Physik*, **12**, 593 (1931).

[8] D. B. Judd, W. N. Harrison, and B. J. Sweo, "Optical Specification of Vitreous Enamels," *Jour. Amer. Ceram. Soc.*, **21** [1] 16–23 (1938).

to the thickness of the ring. A reflectance reading was taken, the thickness of the sample checked, and the reading was repeated. A clear glass plate was used on top of the sample to give a smooth upper surface and a uniform thickness to the layer containing the opacifier. A correction was then made in the readings for the effect of the glass plate. The arrangement of the test sample is shown in Fig. 2.

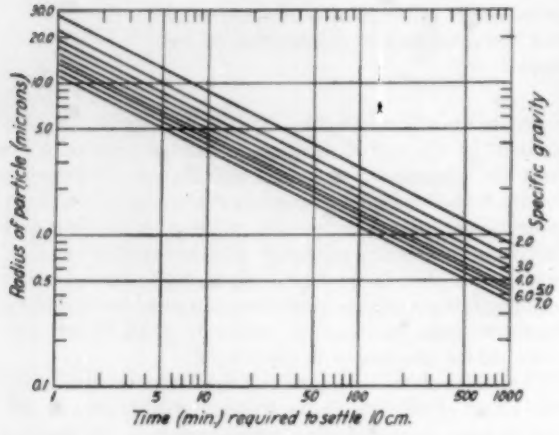

FIG. 1.—Rate of settle in water at 20°C. for particles of various sizes and densities.

$$t = \frac{765}{r^2(s-1)}$$

t = time (min.).
r = radius of particle (μ).
s = sp. gr. of particle.

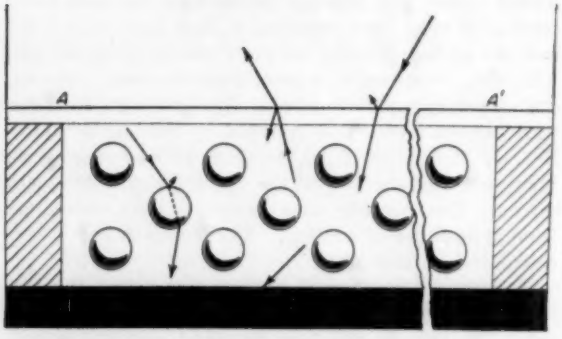

FIG. 2.—Arrangement of test sample.

The cover glass, glycerine, and black glass base had practically the same index of refraction, but the absorption by the cover glass and its thickness affected the reflectance reading. This can be corrected by determining the effect on enamel plates of known reflectance when readings are taken with the enameled sheets wetted with glycerine and covered with the clear glass plate. The glycerine is used to eliminate reflection from the lower surface of the glass and the upper surface of the enamel. The effect may be seen in Fig. 2. By using a series of enamel plates of known reflectance, a calibration curve was obtained, such as is in shown Fig. 3.

III. Data

The data given in Tables I and II were obtained by the method outlined.

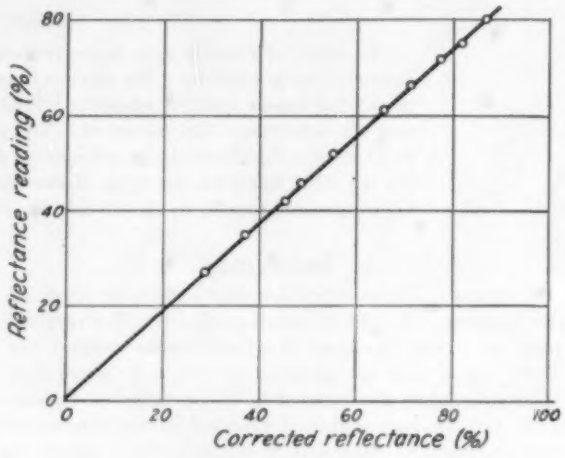

FIG. 3.—Calibration curve for glass No. 3.

TABLE I

REFLECTANCE DATA (GREEN LIGHT)

(For 5-mil layer on a black glass background)

5% zircon ($ZrSiO_4$) in 95% glycerine (by vol.)

Avg. particle diam. (μ)	0.5	1.5	3.5	7.5	15	25	37.5
Corrected reflectance R (%)	65.0	45.0	33.5	18.8	10.7	7.5	5.3

5% corundum (Al_2O_3) in 95% glycerine (by vol.)

Corrected reflectance R (%)	38.0

5% anatase (TiO_2) in 95% glycerine (by vol.)

Corrected reflectance R (%)	70.0

TABLE II

REFLECTIVITY DATA AND VARIOUS PHYSICAL CONSTANTS

Material	Sp. gr.	Avg. index of refraction (η) $\eta = \sqrt[3]{\omega^2}$	Reflectivity (%)*
Zircon	4.58	1.945	77.5
Corundum	4.00	1.765	80.5
Anatase	3.84	2.533	86.5

* For this determination, a layer about 1/4 inch thick and containing a high concentration of opacifier-glycerine mixture was used.

IV. Analysis of Data

To determine what correlation existed between the size of the zircon particles and their efficiency in scattering light, the average scattering coefficient per unit surface of the particles was calculated in the following manner.

The reflectance, R, in Table I, was first corrected for internal and external reflection at the air-glass interface, $A - A'$ (Fig. 2). For this purpose, an equation used by Ryde[4] was employed. It was first corrected for

the fact that the semitransparent layer was on a black background and that, because of the way in which the reflectometer was constructed, none of the specular reflection from the surface of the layer was included in the reflectance reading. The revised equation is as follows:

$$\rho = \frac{R}{(1 - r_1)(1 - r_2)\, r_2 R} \qquad (1)$$

Wherein ρ = reflectance corrected for internal and external reflections at air-glass interface.
r_1 = external reflection coefficient (0.04).
r_2 = internal reflection coefficient (0.40).
R = reflectance expressed as a ratio instead of in percentage as were values of R in Table I.

The next step was to calculate the scattering coefficient per unit thickness of the semitransparent layer. This was done by using the formula of Kubelka and Munk[7] for such a layer on a black background. Employing equation (1), their formula is as follows:

$$S = \frac{1}{X\left(\frac{1}{\rho_\infty} - \rho_\infty\right)} \ln\left(\frac{1 - \rho\rho_\infty}{1 - \frac{\rho}{\rho_\infty}}\right) \qquad (2)$$

Wherein S = scattering coefficient per unit thickness.
X = thickness of layer.
ρ_∞ = reflectivity corrected for internal and external reflection.

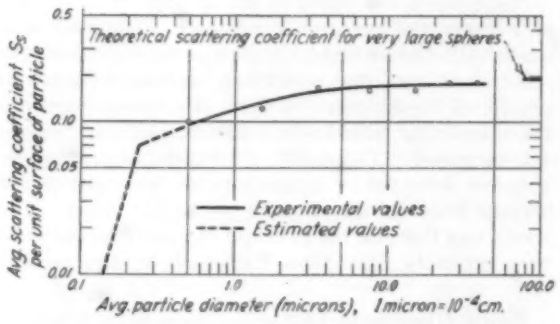

FIG. 4.—Scattering coefficient vs. particle size; particles with average index of refraction of 1.945 in medium with index of 1.470.

The next calculation was the determination of the scattering coefficient, s, per unit surface of the opacifying particles. This was done on the assumption that the backward scattering by a volume of such size as to contain one particle divided by one half the total surface of the particle would give the average scattering coefficient per unit surface of the particle. By "backward scattering" is meant all scattered light leaving a particle in the general direction of the upper surface of the semitransparent layer. The particles were assumed to be spherical and uniformly dispersed. That both assumptions were reasonably justified was verified by microscopic examination.

The calculations were carried out in the following manner. The volume of a cube that will contain one particle, if the opacifying material is uniformly dispersed, will be

$$\frac{\pi D^3}{6} \times \frac{100}{\%\text{ opacifier by volume}}$$

Wherein D = diameter of particle.

This value was multiplied by the scattering coefficient per unit thickness of the enamel layer and divided by one half the area of a single particle. For 5% of opacifying material by volume, the result may be expressed mathematically by equation (3).

$$S_s = \frac{\frac{\pi}{0.3} SD^3}{\frac{\pi D^2}{2}} \qquad (3)$$

$$S_s = 6.67SD.$$

The results calculated by equations (1), (2), and (3) are listed in Table III and are shown in graphical form in Fig. 4.

TABLE III

CALCULATED DATA

Avg. diam. (D) in μ	Corrected reflectance (ρ) calculated from equation (1)	Scattering coefficient (S) per unit thickness in μ calculated from equation (2)	Scattering coefficient (S_s) per unit surface of particle ($S_s = 6.67SD$)
	5% zircon, 95% glycerine by volume		
0.5	0.775	0.03000	0.1000
1.5	0.600	0.01215	0.1215
3.5	0.475	0.00723	0.1685
7.5	0.290	0.00321	0.1605
15.0	0.171	0.001602	0.1602
25.0	0.120	0.001069	0.1780
37.5	0.085	0.000731	0.1828
Reflectivity	ρ_∞ 0.870		
	5% corundum, 95% glycerine by volume		
1.5	0.525	0.01115	0.1115
Reflectivity	ρ_∞ 0.898		
	5% anatase, 95% glycerine by volume		
1.5	0.815	0.03660	0.3660
Reflectivity	ρ_∞ 0.938		

To estimate what maximum reflectance might be expected, the curve in Fig. 4 was extrapolated to a particle diameter of 0.24 micron, the value reported by Stutz[6] as giving the maximum reflectance for green light. By using the corresponding value of the scattering coefficient and by applying equations (3), (2), and (1) in the order named, the reflectance of a layer containing particles of this size could be calculated. This was done, and the reflectance was found to be 70.5%, which was only a little less than the reflectivity (77.5%).

As the particle size is decreased below 0.24 micron, the scattering coefficient per unit surface should decrease rapidly. According to Rayleigh's law of scattering, the coefficient for very small particles should vary as the fourth power of the diameter. Such a curve is shown in Fig. 4, but the change in coefficient is probably not as abrupt as the one indicated.

The experimental and calculated reflectance data are shown in Fig. 5.

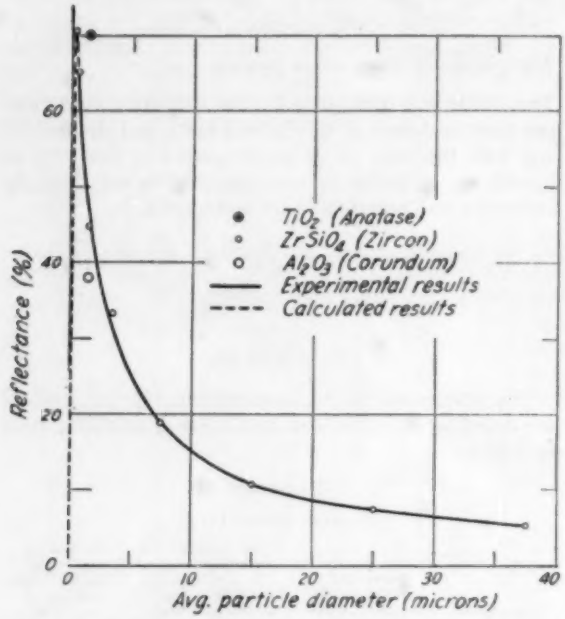

FIG. 5.—Reflectance vs. particle size for 5-mil layer; 5% solids and 95% glycerine by volume

A check on the accuracy of the scattering coefficients of the larger particles is provided by Shoulejkin's calculations of the scattering by large spheres having an index of refraction 1.32 times that of the medium in which they are suspended. This ratio is the same as that between zircon and glycerine. Assuming that the particles are uniformly illuminated by diffuse light of unit intensity, the average scattering coefficient will be the average of the coefficients obtained from zero to 180 degrees. This value is 0.192, which is only slightly higher than the maximum value obtained, namely, 0.1828.

Shoulejkin's equations, which were used in this calculation, are as follows:

$$I_{reflected} = I_{incident}\,\Phi\cos\phi$$

$$I_{refracted} = I_{incident}\,(1-\Phi)\cos\phi$$

Wherein I = intensity of light.

$$\Phi = \frac{1}{2}\frac{\sin^2(\phi-\psi)}{\sin^2(\phi+\psi)}\left[1+\frac{\cos^2(\phi+\psi)}{\cos^2(\phi-\psi)}\right]^2.$$

ϕ = angle of incidence.

ψ = angle of refraction.

V. Discussion

From this investigation, it was found that the particle size of the opacifying material has an important effect on the reflectance obtained from a thin semitransparent layer such as an enamel. The effect is illustrated in Fig. 5, which shows that the reflectance was increased from 5.3 to 65.0% by decreasing the average particle diameter from 37.5 to 0.5 micron. The maximum reflectance was estimated as 70.5% for an average particle diameter of 0.24 micron.

The results were subject to two possible sources of error, one arising from inaccuracies in the determination of the average particle size and the other from nonuniform dispersion of the opacifier throughout the glycerine. The average particle sizes recorded should be reasonably accurate. The zircon dispersed readily in water, and the way in which the various fractions were prepared tended to eliminate those particles that were approaching either upper or lower limits. All of the samples were examined under the microscope, and the particle sizes appeared to be within the limits indicated. There was more chance of error, owing to the nonuniform dispersion of the samples used for the reflectance tests. The particles were mixed until further stirring produced no change in reflectance. There was, however, a tendency for the largest particles to settle out in the glycerine mixture while the reflectance was being measured, making it necessary to take readings as rapidly as possible. The results obtained appear to check very well with the maximum scattering coefficient calculated from Shoulejkin's data. On the whole, the possibilities for error were greatest in the samples containing the smallest particles. The very fine particles were harder to separate, did not disperse as readily in the glycerine, and could not be checked as well by microscopic examination.

Nonuniform dispersion of the opacifier would probably lower the reflectance readings. The two tests made with alumina and titania showed that both the reflectance and the scattering coefficient increased rapidly as the difference in index of refraction between the opacifying particles and the suspending medium was increased. The results with these two materials, however, were not as satisfactory as those with zircon because they did not disperse as well in water. The result was that, on the average, the particles obtained were probably finer than indicated by the settling test.

Figure 4 also shows that as the diameter of a particle is decreased, the ordinary laws governing the reflection and refraction of light gradually break down until, for very fine particles, the only effect tending to change the direction of the light is a relatively weak scattering which is essentially a diffraction phenomenon. The result is that only one size of opacifying material will give the maximum opacity for any given set of conditions. For enamels, the size should lie near $^1/_4$ micron. For particle sizes greater or less than this value, the reflectance is lower, as is shown in Fig. 5.

VI. Conclusions

(1) The particle size and index of refraction of the opacifying material have a decided effect on the reflectance which may be obtained from an enamel for a given amount of opacifier.

(2) The reflectance increases as the difference between the index of refraction of the opacifier and the surrounding medium is increased.

(3) For large particles, the scattering power varies as the total surface of the opacifying material.

Acknowledgment

The author would like to express his indebtedness to A. I. Andrews of the University of Illinois for his suggestions throughout the course of this work and to the Chicago Vitreous Enamel Product Company of Cicero, Illinois, which financed this investigation. This work was done as a project of the Engineering Experiment Station of the University of Illinois.

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REACTIONS TAKING PLACE DURING SMELTING OF SUPEROPAQUE ANTIMONY ENAMEL*

By Burnham W. King, Jr., and Andrew I. Andrews

Abstract

The reactions taking place during the smelting of a typical superopaque antimony enamel were studied by first determining the thermal effects which occur during the smelting of the raw batch and then by investigating the reactions which produce these effects. By this method, it was found that up to 500°F. the chief reaction was the dehydration of the borax. Between 500° and 800°F., the first liquid developed with the melting of the soda niter. Most of the reactions, however, took place from 800° to 1300°F. Soda niter and sodium silicofluoride were decomposed, and the antimony trioxide was oxidized. Above 1300°F., considerable glass was formed, and the enamel gradually became more uniform.

I. Introduction

The object of this investigation was to study the reactions taking place during the smelting of a typical superopaque antimony enamel.

The following batch composition is similar to the one used, except that sodium silicofluoride was substituted for cryolite and antimony was added as Sb_2O_3 instead of $NaSbO_3$:

Very opaque white frit No. 35[1]

	(%)
Feldspar	17.60
Quartz	23.47
Borax	22.88
Soda ash	6.70
Soda niter	2.93
Fluorspar	2.51
Cryolite	8.38
Zinc oxide	2.93
Sodium antimonate	12.58
	99.98

The general procedure followed was first to determine the thermal effects which take place during the smelting of the raw batch and then to investigate the reactions which produced these effects by heating various mixtures of the raw batch constituents to definite temperatures and by examining the quenched samples by X-ray and microscopic technique.

Small batches of about six grams each were tested in this work so that a fairly rapid heating rate could be used and a reasonably uniform temperature for the sample could still be obtained. Thermal effects were studied with a differential Chromel-Alumel thermocouple,† and pure granular aluminum oxide served as the reference material. A 20-cu. cm. aluminum-oxide crucible with a partition in the middle was used to hold the sample and the reference material. The samples were heated in an electric furnace at a rate of approximately 20°F. per minute. A continuous record of weight loss was also made for some of the samples. In order to examine the reactions taking place in the enamel raw batch, a differential thermopile was used which gave a difference in potential between the sample and the standard three times as great as was obtained with a simple differential thermocouple.

II. Data

The differential temperature and weight-loss data are shown in Figs. 1 and 2. The proportions used in the various mixtures were the same as those in the enamel raw batch.

In addition, X-ray pictures were taken of samples of enamel raw batch which had been heated to 800°, 1000°, 1200°, 1400°, 1600°, 2000°, and 2200°F. These

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* Presented at the Forty-Second Annual Meeting, American Ceramic Society, Toronto, Canada, April 9, 1940 (Enamel Division). Received April 9, 1940.

[1] W. N. Harrison, R. E. Stephens, and S. M. Shelton, "Consistency of Eight Types of Vitreous Enamel Frits at and near Firing Temperatures," *Jour. Research Nat. Bur. Stand.*, 20 [1] 39–55 (1938); R. P. 1063; p. 44; *Ceram. Abs.*, 17 [4] 134 (1938).

† The differential thermocouple consisted of two thermocouples with one common lead joining the negative side of each. The junction of one couple was placed in the sample and that of the other in the reference material. The difference in potential between the two positive leads served as a measure of the difference in temperature between the sample and the reference material.

samples were also examined with a petrographic microscope.

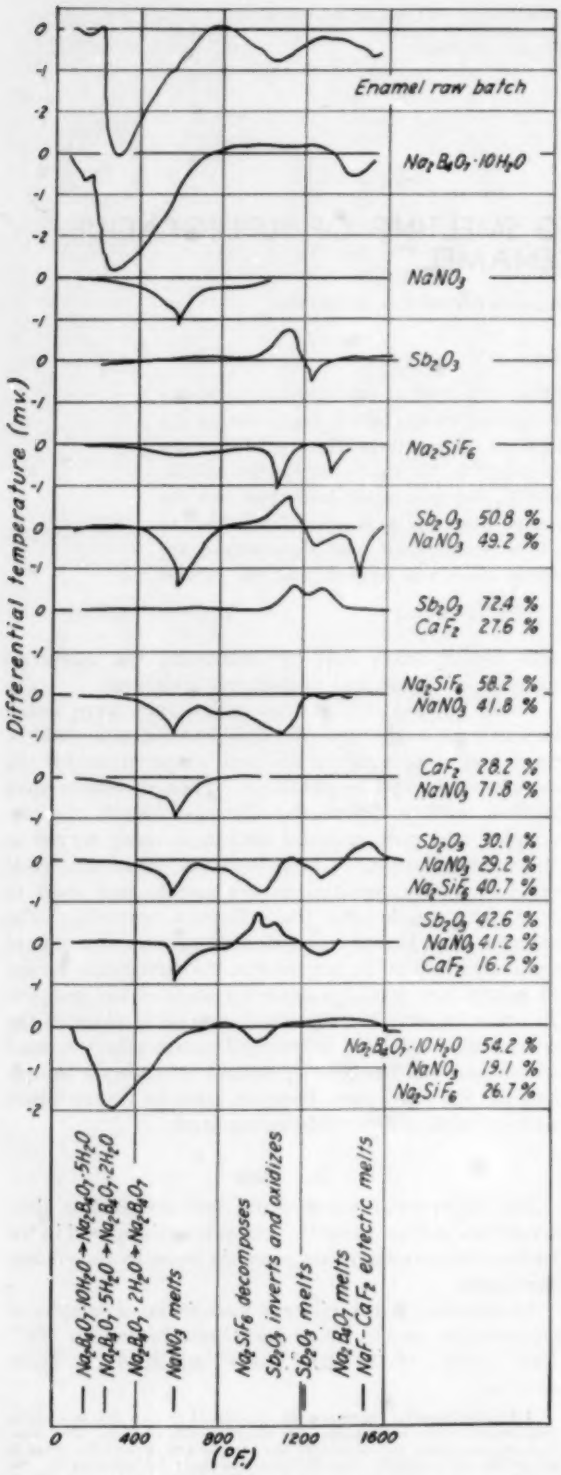

FIG. 1.—Differential temperature.

III. Analysis of Data

From the data obtained, the following reactions were found to take place during the smelting of the enamel investigated.

(1) Room Temperature to 500°F.

On heating a raw batch of enamel, the first reaction noted was the decomposition of the borax. This appeared to take place in two steps, the first occurring at 140° to 150°F. and the second from 230° to about 300°F. According to the literature, $Na_2B_4O_7 \cdot 10H_2O$ decomposes to $Na_2B_4O_7 \cdot 5H_2O$ at 144°F.[2] This corresponds quite closely to the temperature of the first decomposition observed. From the weight-loss data, however, only two molecules of water had been lost up to 230°F., at which point a second decomposition took place. The effect, which was endothermic, reached a maximum at 300°F. This effect corresponds to the reported decomposition of $Na_2B_4O_7 \cdot 5H_2O$ to $Na_2B_4O_7 \cdot 2H_2O$ at 257°F.[2]

The weight-loss data show that at 300°F. the borax had lost about eight of its ten molecules of water. Further decomposition continued over a range of temperatures with the loss of the remaining water of hydration. A slight endothermic effect was noted in the region from 400° to 500°F., the exact temperature varying some with the other materials in the mixture. In the presence of $NaNO_3$, a change was noted in the slope of the differential temperature curve, probably indicating that the rate of dehydration of the borax was accelerated by the presence of this material. In every case, however, dehydration of the borax was practically complete at 500°F.

(2) 500° to 800°F.

The chief effect noted during this period was the marked heat absorption caused by the melting of the sodium nitrate. In the presence of sodium silicofluoride, there was evidence of a reaction between the fused nitrate and the Na_2SiF_6, starting at about 700°F. This was slightly above the temperature at which pure Na_2SiF_6 starts to decompose, namely, at 617°F.[3]

The Sb_2O_3 may oxidize a little during this period, but the effect was negligible for the rate of heating used. The weight loss was relatively small.

(3) 800° to 1300°F.

During this period, the reaction between the Na_2SiF_6 and the $NaNO_3$ continued. When Na_2SiF_6 was heated alone, the rate of decomposition was very slow below 1000°F., but it increased in the presence of $NaNO_3$. The reaction between Na_2SiF_6 and $NaNO_3$ results in the formation of sodium fluoride and silica, which may

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[2] J. W. Mellor, A Comprehensive Treatise on Inorganic and Theoretical Chemistry, Vol. V, p. 124 (1924).

[3] G. Hantke, "Thermische Dissoziation einiger Silicofluoride," *Z. angew. Chem.*, **39**, 1065 (1926); *Ceram. Abs.*, **6** [10] 472 (1927).

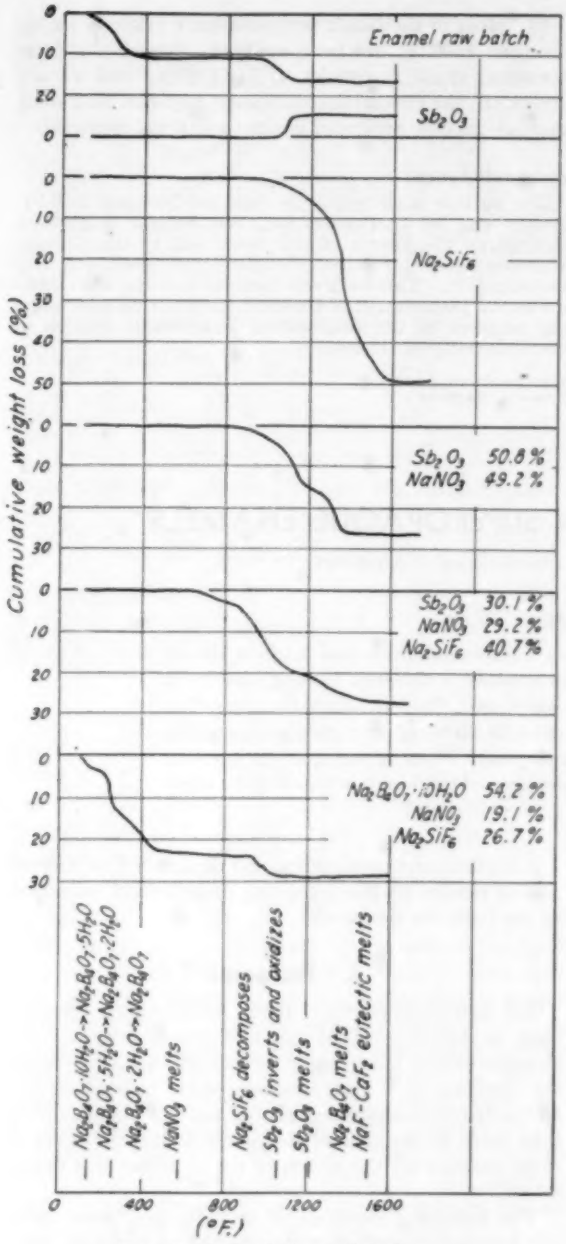

FIG. 2.—Cumulative weight loss.

be shown by an X-ray diffraction pattern of the heated material. In the absence of an excess of soda, the reaction is $Na_2SiF_6 \rightarrow 2NaF + SiF_4$. Silicon tetrafluoride, being a gas, is driven off, and only the sodium fluoride remains.

Two marked endothermic effects were noted in the decomposition of sodium silicofluoride. The first took place at about 1050°F. and the second at 1330°F. The first effect, noted whenever Na_2SiF_6 was present in the mixture, must be related to some change in the form of the original material, although no such change could be found recorded in the literature. The second effect will be discussed later.

Another important effect taking place during this temperature interval was the oxidation of the antimony oxide. If antimony trioxide (senarmontite) is heated in air, it inverts to valentinite at about 1060°F. and then melts at 1210°F.[4] If Sb_2O_3 is heated for several hours at about 1150°F., it oxidizes to approximately Sb_2O_4. On rapid heating, a pronounced exothermic effect took place starting at 1000°F., which indicates that the oxidation or inversion of the antimony trioxide began at about that temperature. The oxidation, however, apparently was not yet complete at 1210°F. as an endothermic effect was still in evidence at the melting point of Sb_2O_3. In the presence of $NaNO_3$, the oxidation was more abrupt and the endothermic effect at the melting point of Sb_2O_3 was much less well defined. The mixture also suddenly lost weight at this temperature, indicating a reaction between the antimony oxide and the $NaNO_3$. What may have happened was that the Sb_2O_3 first started to oxidize slowly, oxygen being taken from the nitrate and added to the Sb_2O_3, thus forming $NaNO_2$ and a higher oxide of antimony. The net weight loss was very small. At about 1170°F., the remaining nitrate, and possibly also the nitrite, suddenly started to decompose further as the weight loss rapidly increased. On continued heating, $NaSbO_3$ was formed at 1350°F., as shown by an X-ray picture.

The presence of CaF_2 altered the differential temperature curve obtained on heating Sb_2O_3 or Sb_2O_3 plus $NaNO_3$. With the latter combination, an antimony compound was formed which had the same crystalline form as Sb_2O_5.

(4) 1300° to 1600°F.

At 1330°F., a marked endothermic effect was noted on heating Na_2SiF_6. According to Hantke,[3] this corresponds to the melting point of $NaF \cdot 4Na_2SiF_6$. The vapor pressure of Na_2SiF_6, however, increases very rapidly in that temperature region, being approximately equal to one atmosphere at 1330°F. The heat required to volatilize the Na_2SiF_6 would alone be enough to account for the marked endothermic effect.

When $NaNO_3$ was present, the effect was small or negligible, as the Na_2SiF_6 apparently had already reacted with the $NaNO_3$ to form NaF and silica.

Any free CaF_2 and NaF form a eutectic melting at 1500°F.

The attack on the feldspar and quartz by the more fluid batch constituents was very rapid during this period. A sample heated for an hour at 1600°F. showed no feldspar and only a few grains of undissolved quartz. This suggests that smelting could be carried out at a much lower temperature provided the whole batch reached the desired temperature.

The weight loss above 1300°F. was very small.

IV. Summary of Results

Room temperature to 500°F.: *dehydration period;* borax is dehydrated.

[4] J. N. Friend, Text of Inorganic Chemistry, Vol. VI, Part V. J. B. Lippincott Co., Philadelphia, Pa.

500° to 800°F.: *development of first liquid;* soda niter melts.

800° to 1300°F.: *reaction period;* soda niter and sodium silicofluoride are decomposed and Sb_2O_3 is oxidized.

1300° to 1600°F.: *glass-forming period;* $Na_2B_4O_7$ melts and combines with quartz and feldspar; CaF_2–NaF eutectic is formed.

V. Conclusions

(1) A large percentage of the heat required for smelting an enamel is used in dehydrating the borax.

(2) Excess Na_2O added as $NaNO_3$ or possibly Na_2CO_3 must be present to prevent excessive loss of fluorine from the Na_2SiF_6.

(3) Most of the batch reactions have taken place by the time 1600°F. has been reached. Above this temperature, small quantities of H_2O, CO_2, and F_2 are driven off, the enamel becomes more uniform, and some changes may be produced in the opacifying material.

Acknowledgment

The authors wish to express their indebtedness to B. T. Sweely and R. L. Fellows for their helpful suggestions throughout the course of this work and to the Chicago Vitreous Enamel Product Company for sponsoring this investigation. This work was done as a cooperative project in the Department of Ceramic Engineering and under the auspices of the Engineering Experiment Station at the University of Illinois.

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DEVELOPMENT OF OPACITY IN SUPEROPAQUE ENAMELS*

BY BURNHAM W. KING, JR., AND ANDREW I. ANDREWS

ABSTRACT

Using X-ray and microscopic technique, a detailed examination was made of the effect of smelting time and temperature on the crystalline material causing opacity in typical superopaque antimony enamels. It was found that, although the crystalline antimony compound causing opacity in such enamels dissolved very slowly at smelting temperatures, there was a decided increase in the particle size of the opacifier particles as the time of smelting was increased. The presence of calcium was found to be beneficial in producing opacity.

I. Introduction

The covering power of an enamel is mainly dependent on (1) the particle size of the opacifying material, (2) the dispersion of the opacifying material, and (3) the difference in refractive index between the opacifying material and the glass which surrounds it. In superopaque antimony enamels, the most important opacifying agent has been reported to have the same crystalline structure as Sb_2O_3.[1]

The object of this investigation was to determine the effect of smelting procedure and enamel composition on the opacity obtained from antimony oxide.

Raw Batch Composition
Frit No. 35[2]

	(%)		(%)
Feldspar	17.60	Fluorspar	2.51
Quartz	23.47	Cryolite	8.38
Borax	22.88	Zinc oxide	2.93
Soda ash	6.70	Sodium antimonate	12.58
Soda niter	2.93		
			99.98

A typical superopaque enamel designated as enamel A and similar to the foregoing composition was used as the basis for this work.

II. Procedure

The investigation was carried out along two general lines as follows: (1) a detailed examination of the changes taking place in the antimony opacifier during the smelting of a typical superopaque enamel similar to the batch composition given, and (2) changes were then made in the enamel composition, and the effect of these changes on the action of the opacifier was determined.

The following methods of investigation were used: (1) Batches of enamel were smelted at different temperatures for varying periods of time, and the reflectance was determined. (2) Very thin-walled bubbles to be used for microscopic examination were blown from the molten enamels just before quenching; these samples were used to determine the particle size and dispersion of the opacifying material. (3) X-ray pictures were taken of the various frits to determine what changes had taken place in the opacifier during smelting. (4) X-ray pictures were also taken of the molten enamel to determine whether or not the opacifying material went into solution during smelting. The equipment used for making this test is shown in Fig. 1. A four-hour exposure was necessary to produce a satisfactory picture.

* Presented at the Forty-Second Annual Meeting, The American Ceramic Society, Toronto, Canada, April 9, 1940 (Enamel Division). Received April 9, 1940.

[1] A. I. Andrews, G. L. Clark, and H. W. Alexander, "Progress Report on Determination of Crystalline Compounds Causing Opacity in Enamels by X-Ray Methods," *Jour. Amer. Ceram. Soc.*, **14** [9] 634–39 (1931).

[2] W. N. Harrison, R. E. Stephens, and S. M. Shelton, "Consistency of Eight Types of Vitreous Enamel Frits at and near Firing Temperatures," *Jour. Research Nat. Bur. Stand.*, **20** [1] 39–55 (1938); p. 44; R. P. 1063; *Ceram. Abs.*, **17** [4] 134 (1938).

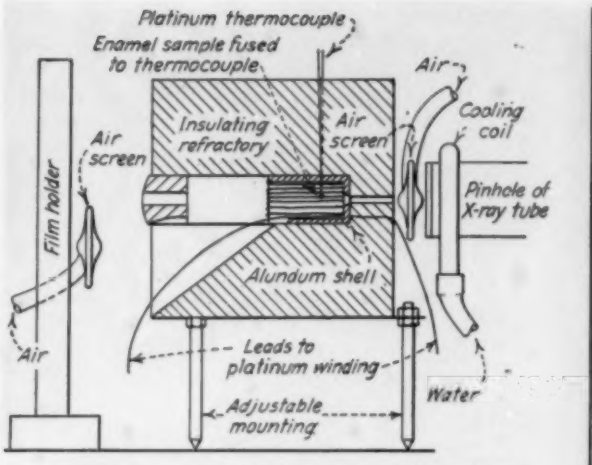

FIG. 1.—High-temperature furnace for X-ray pictures.

III. Data on Enamel A

(1) Properties

The effect of the smelting schedule on reflectance is given in Table I.

TABLE I

Reflectance at 64 Gm./Sq. Ft.

Temp. (°F.)	Smelting time (hr.) 1/2	3/4	1	2	3	5	10
2000	68.0	70.0	71.5	75.5	75.6	76.0	74.5
2100	71.5	74.0	75.0	75.0	75.0	73.5	
2200	70.0	75.0	72.5	69.0	67.5	67.0	

All enamels were prepared in an electric smelter and were fired for three minutes at 1500°F. in an electric furnace. Clay and magnesium carbonate were added at the mill, but no opacifier was used.

Photomicrographs of some of the thin-walled bubbles blown from the molten enamels are shown in Fig. 2. These pictures were taken with transmitted light and at a magnification of 500 diameters. Except for the undersmelted enamel, No. 1, all of the enamel bubbles had nearly the same wall thickness. The smelting schedule and the reflectance of each enamel for an application of 64 gm. per sq. ft. are given.

The effect of the time of smelting on the particle size of the antimony opacifier is shown in Table II.

TABLE II

EFFECT OF SMELTING TIME ON OPACIFIER PARTICLE SIZE

Smelting temp. 2200°F.

Smelting time (hr.)	1	1 1/2	2	4	6	8 1/4
Estimated avg. particle diam. (micron)	0.25	0.50	0.75	1.5	2.0	2.5

The X-ray pictures shown in Fig. 3 are typical of undersmelted, correctly smelted, and oversmelted enamels. The change from smooth lines in the X-ray

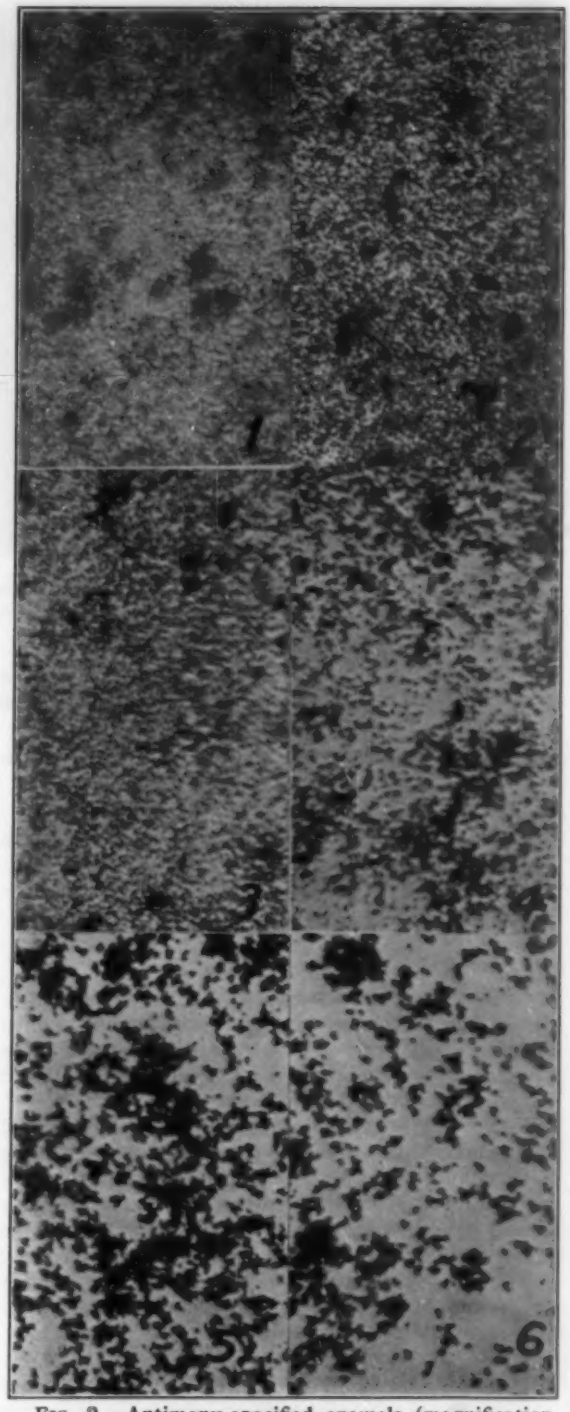

FIG. 2.—Antimony-opacified enamels (magnification 500 × with transmitted light): (1) smelted 1 hour at 2000°F., reflectance 71.5% at 64 gm. per sq. ft.; (2) smelted 3 1/2 hours at 2000°F., reflectance 75.6% at 64 gm. per sq. ft.; (3) smelted 1 hour at 2200°F., reflectance 72.5% at 64 gm. per sq. ft.; (4) smelted 2 hours at 2200°F., reflectance 69.0% at 64 gm. per sq. ft.; (5) smelted 5 hours at 2200°F., reflectance 67.0% at 64 gm. per sq. ft.; (6) smelted 8 1/4 hours at 2200°F., reflectance 66.5% at 64 gm. per sq. ft.

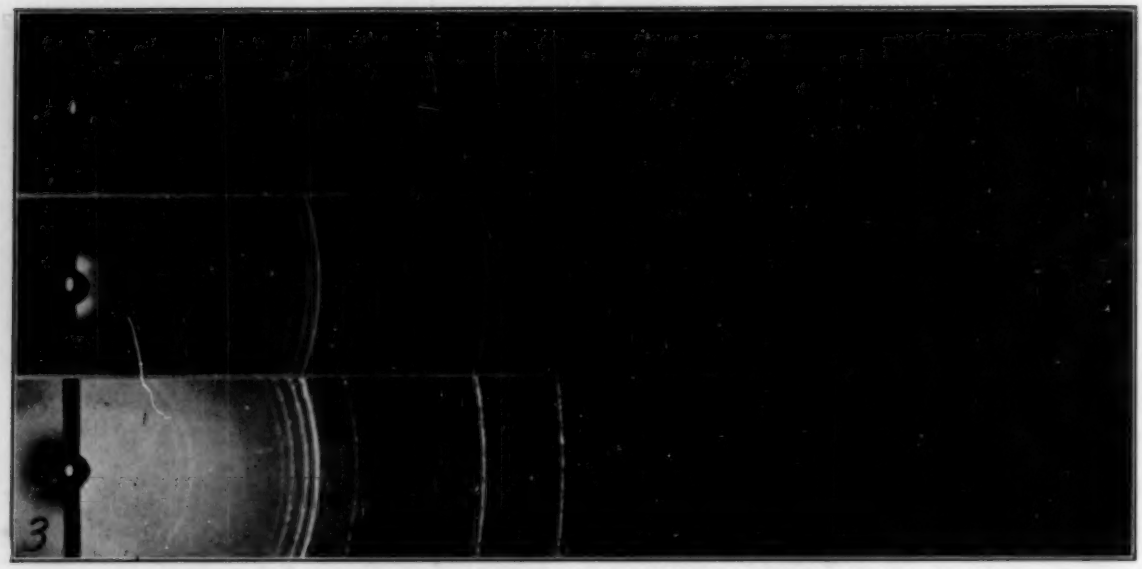

FIG. 3.—X-ray patterns of antimony-opacified enamels: (1) undersmelted, (2) correctly smelted, and (3) oversmelted.

TABLE III
EFFECT OF CHANGES IN COMPOSITION

Enamel No.	A	B	C	D	E
Change from enamel A	None	No calcium	No fluorine	No antimony	No $NaNO_3$
Reflectance at 64 gm./sq. ft.	72.5	50.5	Very low	47.5	55.0
Crystalline materials (shown by X-ray pattern)	Sb_2O_5 (strong); NaF (weak)	Sb_2O_5 (very weak)	Identity in doubt	NaF (weak); CaF_2 (weak)	Sb_2O_5 (very weak); NaF (weak)
Antimony oxide (particle size)	Small and uniform	Variable			Variable

pattern for the undersmelted enamel to ragged or dotted lines in the pattern for the oversmelted enamel indicates that there was a decided increase in the particle size of the opacifying material as the time or temperature of smelting was increased.

X-ray pictures taken at high temperatures showed that under usual smelting conditions there is little likelihood that the antimony opacifier completely dissolves at smelting temperatures and recrystallizes during the subsequent quenching and firing. After the enamel had once been fritted, the crystalline material present at 1830°F. was identical with that present at room temperature except for a small quantity of sodium fluoride.

(2) Effect of Composition of Properties

The changes in composition producing the most decided effects in the properties of enamel A as well as reflectance and X-ray data are given in Table III. All of these batches were smelted for one hour at 2200°F.

From these results, it appeared that calcium, fluorine, and $NaNO_3$ were necessary to produce good opacity with antimony. It was then decided to see what compounds would be produced if a mixture of these materials were calcined. Mixtures containing various proportions of CaF_2, $NaNO_3$, and Sb_2O_3 were prepared. X-ray pictures were made of the calcines, which were also used as raw batch additions. The X-ray pictures showed a decided decrease in the crystalline fluorspar present in the calcined mixtures as compared with the amount added in the original batches. A chemical analysis* of a washed sample of the calcine giving the best opacity was approximately $CaO \cdot 2CaF_2 \cdot 3Sb_2O_{3-5}$. This material has the same crystalline structure as Sb_2O_5.

The properties of a calcium-free enamel (enamel B, Table III) were investigated further. Various smelting procedures were tried, but the reflectance of this enamel was never as good as that obtained for enamel A. Microscopic examination of thin films of this enamel showed a decided difference in the appearance of the opacifying particles. Photomicrographs of two samples of enamel B are shown in Fig. 4.

* Chemical analysis made by the Chicago Vitreous Enamel Product Co.

The dispersion of the opacifier in these samples was evidently not nearly so good as when calcium was present, and there appeared to be less crystalline material present. To estimate the relative amount of solution in the different enamels, the quantity of crystalline material present was estimated by a particle count and was checked by determining the change in the index of refraction of the enamel glass. In making this estimate, the antimony oxide in the glass was assumed to have the same density and index of refraction as it would in a mineral. The calculations were made on the basis of the theoretical melted composition, neglecting volatilization losses. The results are given in Table IV.

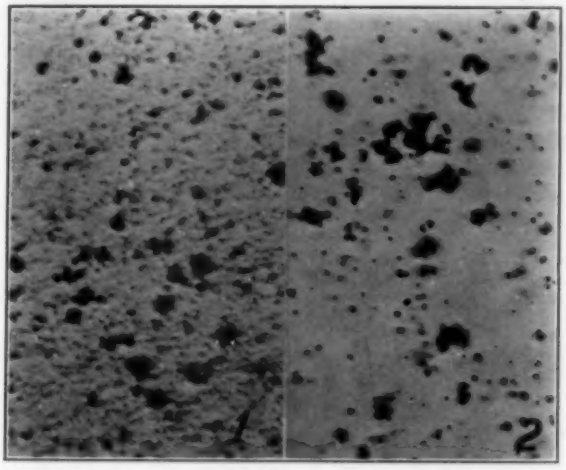

FIG. 4.—Antimony-opacified enamels which do not contain calcium (magnification 500 × with transmitted light): (1) smelted 1 hour at 2000°F., reflectance 64.5% at 64 gm. per sq. ft.; (2) smelted 1 hour at 2200°F., reflectance 50.5% at 64 gm. per sq. ft.

TABLE IV

AMOUNT OF CRYSTALLINE MATERIAL IN VARIOUS ENAMELS

Enamel No.	Smelting time (hr.) at 2000°F.	Amount of antimony oxide undissolved (% by weight) Based on microscopic count	Based on change in index of glass	Reflectance at 64 gm./sq. ft.
A	3½	Particles too fine	No data	75.6
A	8¼	Particles too fine	84	66.5
B	1	83	76	50.5
B	5¼	34	36	Very low

IV. Discussion

When a typical superopaque enamel is oversmelted, the loss of opacity due to the solution or volatilization of antimony oxide is relatively small. The chief reasons for the loss in opacity are the increase in the particle size of the opacifying material and the decrease in the uniformity with which it is dispersed through the enamel glass. The importance of the effect of particle size on the opacity obtained from thin layers such as enamels was stressed in a previous paper.[3]

The individual opacifier particles are very small in an undersmelted enamel, but they are collected together in large loose aggregates known as antimony spots. As smelting is continued, these aggregates tend to break up, and the dispersion of the opacifier improves. At the same time, however, the particle size of the individual grains increases either by solution and recrystallization or by coagulation into small dense aggregates. The maximum opacity would be obtained if all the particles were about ¼ micron in diameter[4] and uniformly distributed. In the actual smelting of an enamel, these conditions are only approximated because, by smelting long enough to break up the antimony spots, many particles much larger than the most desirable size are produced. The tendency of the particles to grow and to form dense aggregates on oversmelting is shown by the photomicrographs.

In the absence of lime, there is much less tendency for the large loose antimony aggregates present at low temperatures to break up and disperse through the enamel, as its mobility is increased by rising temperature. Instead, they tend to coagulate and thus form large particles which give poor opacity. The rate of solution of the opacifier was also much more rapid in the absence of calcium so that, on continued smelting, the opacity was further decreased by the loss of some of the opacifying material.

Although no satisfactory quantitative data were obtained for the correctly smelted calcium-bearing enamel, the amount of solution of the opacifier must have been very small as the sample, smelted 8¼ hours or about eleven times the correct smelting period at 2200°F., showed that only 16% of the antimony oxide had been dissolved.

The difference in properties caused by the presence of calcium is probably due to the formation of a crystalline phase which contains calcium, antimony, oxygen, and fluorine, such as was found in calcined mixtures of these materials. Inasmuch as it has the same crystalline structure as Sb_2O_5, it may be a solid solution of CaO or CaF_2 or of both in Sb_2O_5. This material appears to be more inert, as it shows less tendency to coagulate than Sb_2O_5 or Sb_2O_4, which is probably formed in the absence of calcium and is less soluble in the enamel glass.

V. Conclusions

(1) The variations in the opacity of superopaque antimony enamels caused by changes in the smelting treatment are mainly due to variations in the particle size and dispersion of the opacifying material. In general, the tendency is for the opacifier particles to grow and to coagulate as either the time or temperature of smelting is increased. This tendency is illustrated by the photomicrographs in Fig. 2.

(2) The amount of antimony oxide, which is dissolved

[3] B. W. King, Jr., "Effect of Particle Size and Index of Refraction on Reflectance," *Jour. Amer. Ceram. Soc.*, this issue, pp. 225–28.

[4] G. F. A. Stutz, "Scattering Light by Dielectrics of Small Particle Size," *Jour. Franklin Inst.*, **210** [1] 67–85 (1930); *Ceram. Abs.*, **10** [4] 306 (1931).

or volatilized during smelting under ordinary conditions, is relatively small provided sufficient calcium is present.

(3) The crystalline antimony compound causing opacity in typical superopaque enamels is probably composed of calcium, antimony, oxygen, and fluorine and has the same crystalline structure as Sb_2O_5.

Acknowledgment

The authors wish to express their indebtedness to B. T. Sweely and R. L. Fellows for their helpful suggestions throughout the course of this work and to the Chicago Vitreous Enamel Product Company for sponsoring this investigation. They also wish to thank G. L. Clark of the Analytical Division, University of Illinois, for the use of the X-ray equipment. This work was done as a cooperative project in the Department of Ceramic Engineering and under the auspices of the Engineering Experiment Station at the University of Illinois.

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VISCOUS PROPERTIES OF SODA-BORIC OXIDE-SILICA GLASSES AT TEMPERATURES FROM 900° TO 1500°F.*

By Burnham W. King, Jr., and Andrew I. Andrews

Abstract

The viscosities of a number of soda-boric oxide-silica glasses were approximately determined in the temperature range from 900° to 1500°F. These were obtained by noting the deformation of small glass rods. Three steps were observed in the process of deformation which occurred at definite viscosities, the absolute magnitudes being obtained by correlation with glasses of known properties.

I. Introduction

Although many enamels contain from 65 to 90% of sodium oxide, boric oxide, and silica, there are few data available on the viscous properties of the glasses formed from these oxides in the range of compositions and temperatures of interest in enameling. Inasmuch as these three materials constitute such a large part of the melted composition of most enamels, they control to a large extent the properties of the enamel glass. Other additions, such as fluxes, opacifiers, and coloring oxides, may be considered to modify (to a greater or lesser degree) the properties contributed by the three important constituents, namely, soda, boric oxide, and silica. A knowledge of the properties of the glasses formed from these materials makes it possible to tell what may be expected of enamels based on these relatively simple compositions. The viscosity of enamels is of interest because this property is probably the most important single factor in determining whether or not an enamel will smooth out, heal over defects, and develop good gloss during firing. The object of this investigation was to make a general survey of this field of compositions, using tests that could be made rapidly and yet would yield satisfactorily comparative data.

II. Procedure

The compositions studied along with some of those investigated by other authors are shown on the triaxial diagram in Fig. 1. The compositions indicated for this investigation are on the basis of the theoretical melted compositions as calculated from the raw batches. The batch constituents were quartz, soda ash, and boric acid. Fifty-gram batches were weighed, placed in fireclay crucibles, and melted in an electric furnace at 2200°F. The crucibles were removed from the furnace, and rods were drawn from the glasses as they cooled. These rods were used to determine the temperatures at which various degrees of softening took place.

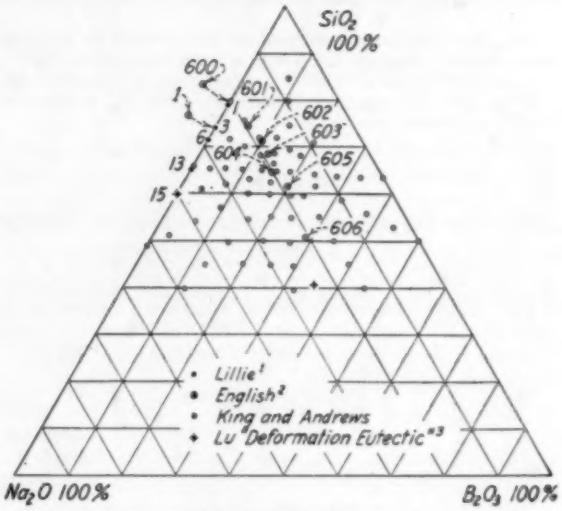

Fig. 1.—Compositions studied by various authors.

* Presented at the Forty-Second Annual Meeting, The American Ceramic Society, Toronto, Canada, April 11, 1940 (Enamel Division). Received April 11, 1940.

[1] H. R. Lillie, "High-Temperature Viscosities of Soda-Silica Glasses," *Jour. Amer. Ceram. Soc.*, **22** [11] 367–74 (1939).

[2] S. English, "Effect of Composition on Viscosity of Glass, II," *Jour. Soc. Glass Tech.*, **8** [31] 205 (1924); *Ceram. Abs.*, **4** [1] 5 (1925).

[3] Kai-Ching Lu, "Deformation Study of Various Aluminosilicates and Borosilicates," *Jour. Amer. Ceram. Soc.*, **9** [1] 29–54 (1926).

Test No. 1 consisted in determining the temperatures at which rods, 1 cm. long and 0.75 mm. in diameter, would draw up into a bead or elongate and break under their own weight when they were suspended from one end and heated in a furnace for five minutes.

Test No. 2 was made to determine the temperatures at which rods, 3.25 cm. long and 0.50 mm. in diameter, mounted at a 60-degree angle in a rigid base and heated for one hour, would bend over and flatten out so that the rod touched the base along its whole length.

In the test No. 3, the temperatures were determined at which similar glass rods, mounted in the same way, would bend until their tips just touched in one hour.

In order for a glass rod to deform in the manner indicated in any one test, it was necessary for it to have a certain viscosity. The absolute values of the viscosities indicated by the three different tests were determined by a comparison with soda-boric oxide-silica glasses of known viscosities.

The results of this comparison given in Table I show that the average viscosities for the degree of softening noted in tests (1), (2), and (3) were, respectively, 4.0×10^5, 6.6×10^7, and 1.7×10^{10} poises.

In the test method used for the two higher viscosities, essentially a cone fusion test, a plaque was made to hold all of the samples at one time and in the same positions as those in which they are shown in Fig. 1. The tests were made by first placing the plaque in a sagger in an electric furnace. In about twenty minutes, the temperature was raised to the desired point, held for one hour, and the samples were then quickly removed. Check runs were made with the glass rods in various positions on the plaque, but the temperature distribution was sufficiently uniform to cause the rods to deform in the same order irrespective of their relative positions on the plaque. The tests, which depended on the glass rods elongating rapidly or drawing up into beads, were performed in an electric enameling furnace. The rods to be tested were first beaded on one end and then suspended in the loops of a wire rack. This rack was placed in the furnace, heated for five minutes, quickly removed, and the results were noted.

III. Data

The temperatures at which the different glasses had viscosities of 4.0×10^5, 6.6×10^7, and 1.7×10^{10} are shown, respectively, in Figs. 2, 3, and 4. Table I shows the viscosities of the known glasses used in calibrating the experimental results. In using the data of English, a correction was made in the values obtained for the higher viscosities. Stott[4] has shown that such a correction is necessary. Figure 5 shows the plaque and the glass rods after they have been heated for one hour at 1100°F.

IV. Discussion

The methods used in this investigation were such as to give only comparative results. There were several

[4] V. H. Stott, "Viscosity of Glass," *Jour. Soc. Glass Tech.*, **9** [35] 207–25 (1925); *Ceram. Abs.*, **5** [2] 47 (1926).

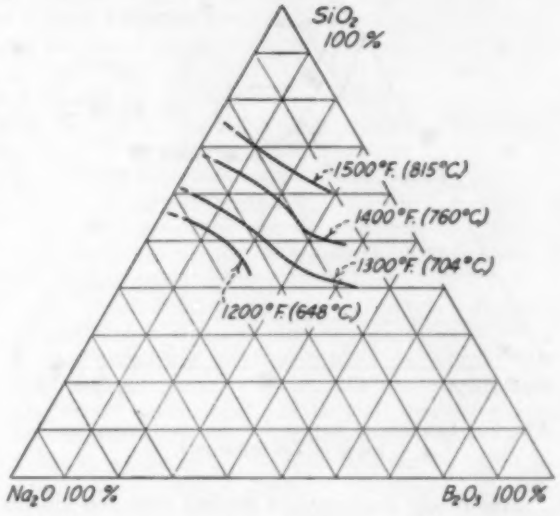

FIG. 2.—Isothermal curves for constant viscosity (η); $\log \eta = 5.6$ (approx.).

TABLE I

CALIBRATION OF SOFTENING EFFECTS IN TERMS OF VISCOSITIES*

Column (1), S. English composition No.; (2) temp. °C. at which glass rod will bend and touch (test No. 3); (3) log of viscosity of glass rod from English data; (4) temp. °C. at which glass rod will flatten (test No. 2); (5) log of viscosity of glass rod from English data; (6) temp. °C. at which glass rod will draw up into bead or elongate rapidly (test No. 1); (7) log of viscosity of glass rod from English data; (8) log of viscosity of glass rod from Lillie data; (9) Lillie composition No.

(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)
600	(585)†	9.8	(755)†	6.3	(840)†	5.2	5.0	1
1	525	10.6	630	7.8	800	5.3	5.1	3
601	(630)†	9.5	(760)†	6.6	(825)†	5.4		
602	(640)†	9.5	(740)†	6.9	(820)†	5.5		
603	640	10.0	730	7.4	815	5.3		
604	640	10.1	720	7.7	785	5.8		
605	640	10.0	710	7.7	780	5.7		
606	610	10.4	665	8.5	755	5.7		
					770		(5.2)†	6
					730		(5.2)†	13
					690		(5.3)†	15
Avg. (omitting estimated values)		10.22		7.82		5.60	5.1	
Avg. (poises)		1.66×10^{10}		6.61×10^7		3.98×10^5		

* For the references to the work of English and Lillie, see footnotes 1 and 2 in Fig. 1.
† Estimated values.

sources of error. First, data obtained on glasses of compositions as calculated from the raw batches wee compared with data obtained on glasses of known composition. The error from this source was probably small, as the batches were never heated for more than six hours at 2200°F.; therefore any changes in composition due to volatilization or crucible attack should not have been very great. Second, another source of error, encountered in the determination of the lowest value of viscosity, was the variations in the surface tension of the glasses with changes in composition. From the published data on surface tension,[5] an error of about ±10% is to be expected. This error, however, is relatively small when viscosities are being considered.

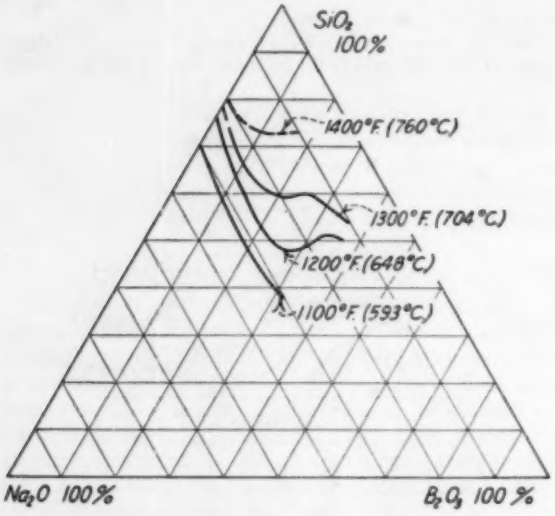

FIG. 3.—Isothermal curves for constant viscosity (η); $\log \eta = 7.8$ (approx.).

Some of the compositions studied were too viscous to melt down to uniform glasses in six hours at 2200°F. This was true of those compositions approximating English's compositions, Nos. 600, 601, and 602, so that the values given for these glasses in Table I could only be approximated. A few glasses which were low in soda and high in boric oxide could not be drawn into smooth threads although they fined readily. Some of the glasses which were high in soda devitrified so rapidly that no satisfactory results were obtained.

As seen from Figs. 2, 3, 4, and 5, it appears that for the range of compositions studied, soda is usually a more powerful flux than boric oxide, although this is not always the case.

At relatively high temperatures (Fig. 2), the substitution of boric oxide for silica produced a more fluid glass. On the other hand, at lower temperatures (Figs. 3 and 4) when boric oxide was added in a like manner, it first increased and then decreased the viscosity. The proportions of boric oxide and silica giving the maximum viscosity varied with the soda content, more boric oxide being required for the glasses higher in soda. For those glasses containing approximately from 55 to 75% of silica and from 10 to 20% of soda, variations in composition had little effect on the viscosity in the range from 10^5 to 10^8 poises (Figs. 3 and 4). When the soda was increased to more than 20%, however, a sudden decrease in the viscosity was noted (Fig. 5).

English,* in working with glasses containing 20% of soda, studied glasses along the edge of this region. He observed a definite maximum in viscosity at about 15% of boric oxide when the glasses were at temperatures below 750°C. (1382°F.). As the constant viscosity surface, shown by the contours in Figs. 3 and 4, is convex at low temperatures, it is possible to vary the glass compositions along a number of different lines and still obtain maxima, such as were observed by English. It should be noted, however, that the soda and boric-oxide proportions at which the maxima occur may vary considerably, depending on the range of compositions studied.

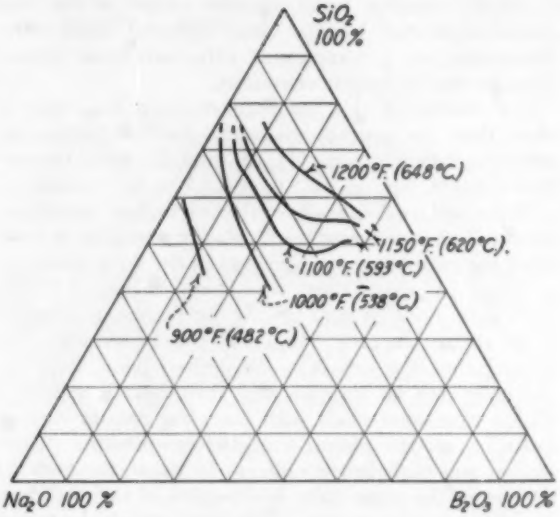

FIG. 4.—Isothermal curves for constant viscosity (η); $\log \eta = 10.2$ (approx.).

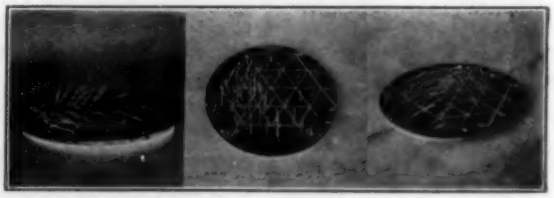

FIG. 5.—Glass rods heated 1 hour at 1100°F.

Information on firing range may also be obtained from these data. The firing range usually should vary as the temperature increase required to change the

[5] C. W. Parmelee, K. C. Lyon, and C. G. Harman, "Surface Tensions of Molten Glass," *Univ. of Ill. Eng. Expt. Sta. Bull.*, No. 311, pp. 5–47 (1939); *Ceram. Abs.*, **18** [11] 295 (1939).

*See footnote 2 in Fig. 1.

viscosity from 10^{10} to 10^{6} poises. The greater the temperature interval, the longer the firing range. On this basis, the usual effect of the addition of boric oxide was to shorten the firing range, the action being more pronounced for glasses high in soda.

Acknowledgment

The authors wish to express their indebtedness to B. T. Sweely and R. L. Fellows for their suggestions throughout the course of this work and to the Chicago Vitreous Enamel Product Company for sponsoring this investigation. This work was done as a cooperative project in the Department of Ceramic Engineering and under the auspices of the Engineering Experiment Station at the University of Illinois.

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SHORT METHODS FOR CHEMICAL ANALYSIS OF GLASS*

By P. A. Webster and A. K. Lyle

Abstract

Analytical methods have been developed which shorten the time required for the determination of many of the constituents found in glass. These short methods have been worked into a procedure for the routine chemical analysis of container and sheet glasses.

The determination of SiO_2, BaO, SO_3, R_2O_3, CaO, MgO, B_2O_3, Na_2O, and K_2O percentages on several samples can be made in a single day. Most of the experimental work has been done on Bureau of Standards glass No. 128, but results from several other glasses are included. Particular care has been taken to discover interfering substances and to eliminate errors from this source. The accuracy is comparable with that obtained from the use of umpire methods.

I. Introduction

There have been several notable advances in analytical chemistry that are of particular interest to silicate analysts, especially to those engaged in the analysis of glasses and raw materials used in glass manufacture. Among the reagents that have been welcomed in recent years are 8-hydroxyquinoline,[1] used for magnesia and sometimes for alumina; zinc uranyl acetate,[2] for the direct determination of soda; and perchloric acid, which holds some advantages over hydrochloric or sulfuric acids for several operations. The direct determination of potassium by the Hicks method,[3] the partition method for boric acid,[4] and some of the shortened methods for the separation of silica involving the use of perchloric acid[5] are also of particular interest to the glass analyst.

The value of all these reagents and procedures in analytical chemistry has already been established. It was the purpose of this investigation to combine these separate methods into a complete procedure for the analysis of commercial bottle and window glasses. After numerous trials and modifications of these shorter methods, a scheme has been worked out for all of the ingredients commonly present. Fundamentally, the procedure is a unit system in which, if desired, a separate sample or aliquot may be used for each constituent. Thus, after volatilizing silica, it is possible to make direct determinations of barium, aluminum, calcium, magnesium, sodium, potassium, and sulfate on separate aliquots, with little preliminary treatment. Silicon and boron are also determined by short methods after fusion with sodium carbonate.

II. Outline of Procedure

Silica is separated from a half-gram sample of crushed glass by fusion with 1 gram of sodium carbonate, followed by decomposition and a single dehydration in perchloric acid. The determination is completed by the usual filtration, ignition, and volatilization.

Barium, aluminum, calcium, and magnesium are separated in turn from a 1-gram aliquot portion of a 2-gram sample that has been decomposed by hydrofluoric-perchloric acid treatment. Complete elimi-

* Presented at the Forty-First Annual Meeting, American Ceramic Society, Chicago, Ill., April 18, 1939 (Glass Division). Received March 20, 1940.

[1] H. B. Knowles, "Use of 8-Hydroxyquinoline in Determinations of Aluminum, Beryllium, and Magnesium," *Jour. Research Nat. Bur. Stand.*, **15** [1] 87–96 (1935); R.P. 813; *Ceram. Abs.*, **14** [11] 294 (1935).

[2] (*a*) H. H. Barber and I. M. Kolthoff, "Specific Reagent for Rapid Gravimetric Determination of Sodium," *Jour. Amer. Chem. Soc.*, **50** [6] 1625–31 (1928).

(*b*) F. W. Glaze, "Direct Determination of Soda in Soda-Lime Glasses by Precipitation as Uranyl Zinc Sodium Acetate," *Jour. Amer. Ceram. Soc.*, **14** [6] 450–53 (1931).

(*c*) E. W. Koenig, "Direct Determination of Sodium Oxide in Feldspar," *ibid.*, **22** [1] 24–31 (1939).

[3] W. B. Hicks, "Rapid Modified Chloroplatinate Method for Estimation of Potassium," *Jour. Ind. Eng. Chem.*, **5**, 650–53 (1913).

[4] F. W. Glaze and A. N. Finn, "Routine Determination of Boron in Glass," *Jour. Research Nat. Bur. Stand.*, **16** [5] 421–29 (1936); R.P. 882; *Ceram. Abs.*, **15** [8] 234 (1936).

[5] H. H. Willard and W. E. Cake, "Perchloric Acid as a Dehydrating Agent in Determination of Silica," *Jour. Amer. Chem. Soc.*, **42**, 2208 (1920).

nation of fluorine is insured by a final treatment with boric acid.

Barium is precipitated as sulfate, and after a brief digestion is separated by filtration, ignited, and weighed.

Alumina is precipitated in the filtrate with ammonia and may be filtered out, ignited, and weighed. If a volumetric procedure is preferred, the hydroxide precipitate may be dissolved in acid, reprecipitated with 8-hydroxyquinoline, and titrated bromometrically.

Calcia is separated as the oxalate from the filtrate from the alumina precipitation and is determined by the usual permanganate titration.

Magnesia is determined in the calcia filtrate by precipitation as quinolate, followed either by titration or by gravimetric estimation.

Soda is precipitated from a separate aliquot, representing 0.1 gram of glass, by direct precipitation as sodium zinc uranyl acetate. The precipitate is dried and weighed.

Potassium is separated directly as the chloroplatinate from another half-gram aliquot. After thorough washing, the potassium precipitate is redissolved, and the platinum is precipitated by reduction with magnesium. The determination is completed by filtering, igniting, and weighing the platinum.

Sulfate is determined on a separate 3-gram sample dissolved by hydrofluoric and perchloric acid treatment. The sulfate is precipitated from a hot solution by the addition of barium chloride.

Boric oxide is determined on a separate 1-gram sample fused with 2 grams of sodium carbonate by a "partition" method, based on that described by Glaze and Finn.[4] This method involves solution of the carbonate melt in water, acidifying, and diluting to 25 ml., followed by extraction of a definite part of the boric acid with a mixture of alcohol and ether. Evaporation of an aliquot portion of the alcohol-ether layer and re-solution in water permits a simple titration of the boric acid with standard alkali and mannite.

III. Detailed Procedure

(A) Silica

Weigh out exactly 0.5000 gm. of powdered glass, transfer to an agate mortar, and mix intimately with 1 gm. of sodium carbonate by careful grinding. Transfer the mixture quantitatively to a 75-ml. platinum evaporating dish, heat first with a small flame, then with the full flame of a blast burner until a clear melt is obtained. Allow the melt to solidify in a thin layer covering the bottom of the dish. Add about 10 ml. of 1:1 nitric acid and place the dish (covered) on a steam bath until all of the carbonates are decomposed. Remove, rinse the cover glass with a small quantity of water, and allow the solution to evaporate to dryness. Add 10 ml. of 60% perchloric acid, and transfer the dish to a hot plate at about 200° to 220°C. Heat until fumes are evolved freely, then cover with a watch glass, and allow fuming to continue for 20 minutes. Remove the dish, cool, and dilute the contents with about 50 ml. of hot water. Stir to dissolve soluble salts, and place dish on steam bath for a 10-minute digestion period. Pour the supernatant solution through a No. 589 S & S blue ribbon filter paper, or equivalent, and flood the residue with about 10 ml. of 1:1 hydrochloric acid. Stir and grind the solid material with a glass rod; then pour the solution through the filter. Transfer the precipitate completely to the filter by means of hot 1:99 hydrochloric acid, using a rubber policeman, and continue washing the paper and precipitate with 8 or 10 small portions of the dilute acid. Place the paper and precipitate in an untared 20-ml. platinum crucible, add 3 or 4 drops of 1:1 sulfuric acid, and ignite slowly in a muffle furnace until the residue is white. Cover and heat over a blast flame for 20 minutes, cool in a desiccator, and weigh. Moisten the residue with water, add about 10 ml. of hydrofluoric acid and 1 drop of sulfuric acid, and evaporate to dryness on a hot plate. Cover the crucible and heat over a blast flame for 1 or 2 minutes. Cool in a desiccator, and weigh. The loss in weight represents silica.

$$\text{Wt. of } SiO_2 \times 200 = \%\ SiO_2.$$

(B) Barium, "Iron and Aluminum," Calcium, Magnesium, Sodium, and Potassium Oxides

DECOMPOSITION OF SAMPLE

Weigh 2 gm. of powdered glass (finer than 50-mesh) into a 75-ml. platinum dish, moisten with water, add 1 ml. of concentrated nitric acid, 10 ml. of 60% perchloric acid, and about 25 ml. of 48% hydrofluoric acid. Evaporate on a steam bath to small volume. remove dish, and add about 10 ml. more of hydrofluoric acid. Again evaporate to small volume. Transfer dish to a hot plate (200° to 215°C.) and allow to fume until about one half the perchloric acid is expelled. Examine for particles of undecomposed glass and, if any are noted, add 5 ml. more of 48% hydrofluoric acid, and again evaporate to copious fumes. Cool the dish, and rinse down the sides with 5 ml. of 60% perchloric acid added from a pipette. Return dish to hot plate, and allow to fume briefly. Cool dish, add about 5 ml. of hot saturated boric acid solution (saturated at 100°C.), and rinse down the sides of the dish with a little distilled water. Again evaporate to fumes, and continue the heating until most of the acid is eliminated but not to the point where the residue appears dry. Add 50 ml. of hot water and 2 ml. of concentrated hydrochloric acid and stir until the soluble salts are entirely dissolved. (The solution may be turbid if both BaO and SO_3 are present.) Rinse into a 200-ml. volumetric flask, cool to room temperature, and dilute to the mark. Mix thoroughly and pipette out the following aliquot portions:

(1) 100 ml. (1 gm.) into a 600-ml. beaker for BaO, "R_2O_3," CaO, MgO.

(2) 10 ml. (0.1 gm.) into a 50-ml. beaker for Na_2O.

(3) 50 ml. (0.5 gm.) into the platinum dish for K_2O.

(1) First Aliquot (BaO, "R_2O_3," CaO, MgO)

(*a*) *Barium Oxide:* Neutralize the 1-gm. aliquot with NH_4OH (methyl red) Then add concentrated hydrochloric acid to 1 ml. excess. Dilute to about

150 ml., cover the beaker, heat to boiling, and add 10 ml. of dilute sulfuric acid (10:90) from a pipette. Continue the boiling for 10 minutes, then transfer to steam bath, and digest for 45 minutes. Filter through a tight paper (S & S No. 589 blue ribbon), transfer the precipitate completely, and wash on the paper with four portions of warm water. Reserve the filtrate and washings for the determination of "R_2O_3." Ignite the precipitate slowly in a tared porcelain crucible in a muffle furnace until the carbon is burned off; then heat at about 1000°C. for 15 minutes. Cool in a desiccator and weigh. (If the precipitate is not white, add a drop of sulfuric acid, heat gently until the acid is vaporized, then again ignite, cool, and weigh.)

Wt. of $BaSO_4 \times 0.657 \times 100 = \%$ BaO.

(*b*) *Iron and Aluminum Oxides:* Heat the filtrate from the BaO determination to boiling, add 1:1 ammonium hydroxide dropwise to about 1 drop excess (methyl red), cover beaker, and allow contents to boil for 2 minutes. Filter immediately through a Whatman No. 41 paper, or equivalent, rinse the beaker twice, and wash the precipitate on the paper four times with small quantities of hot 2% neutral ammonium chloride solution. Reserve the filtrate for determination of CaO.

GRAVIMETRIC METHOD

Ignite the paper and precipitate in a tared platinum crucible, first in a muffle furnace until the carbon is eliminated, and finally for 10 minutes over a blast burner. Cool in a desiccator and weigh.

Wt. of precipitate $\times 100 = \%$ R_2O_3.

VOLUMETRIC METHOD

The "R_2O_3" precipitate, after filtration and washing, may be redissolved in dilute hydrochloric acid, reprecipitated with 8-hydroxyquinoline, and determined by bromometric titration, according to the following procedure:

Return the paper containing the washed hydroxide precipitate to the beaker in which precipitation took place, add a small amount of dilute hydrochloric acid, and stir until the paper is disintegrated. Dilute to 150 ml. Add 10 ml. of 5% 8-hydroxyquinoline solution (see preparation of solutions (under (*d*) *Magnesium Oxide*)), heat to about 60°C., and add ammonium hydroxide dropwise, with stirring, to about a 5-ml. excess. Allow to cool to room temperature. Filter on a 1-G-3 fritted Jena glass crucible with suction, transfer the precipitate completely, and wash four or five times with cold 1:200 ammonium hydroxide. Dissolve the precipitate from the filter by adding 30 ml. of hot 1:1 hydrochloric acid in small quantities, and wash thoroughly with hot water, using suction. Collect the solution in a 500-ml. Erlenmeyer flask. Cool to about 20°C., add 1 gm. of potassium bromide crystals and a few drops of methyl red, and titrate with bromate-thiosulfate exactly as in the MgO procedure. The bromate solution should be standardized against a known alum solution treated exactly in the same manner.

(*c*) *Calcium Oxide:* Heat the reserved filtrate from the "R_2O_3" separation to boiling in a covered beaker and add slowly 20 ml. of saturated ammonium oxalate solution from a pipette. Continue boiling for 5 minutes, transfer to steam bath, and digest for 20 minutes. Filter through a No. 589 S & S white ribbon paper (or equivalent), rinse the beaker thoroughly, and wash the paper and precipitate four times with warm water (60°C.). Reserve the filtrate for determination of MgO. Unfold the paper, and rinse the oxalate precipitate back into the original beaker by means of a stream of warm water. Discard the paper. Dilute to about 100 ml., add 20 ml. of 1:1 sulfuric acid, heat nearly to boiling, and titrate with standard potassium permanganate solution to the first permanent pink.

$KMnO_4$ (ml.) $\times$ CaO factor $\times 100 = \%$ CaO.

Standard Permanganate Solution

Dissolve 44 gm. of potassium permanganate in distilled water, and make up to a volume of 20 liters. This solution should stand for three or four months and may be siphoned off as required. Standardize against a known calcium chloride solution by the above procedure rather than against sodium oxalate.

(*d*) *Magnesium Oxide:* Dilute the filtrate reserved from the CaO separation to 500 ml., add 10 ml. of 5% 8-hydroxyquinoline solution (1 ml. = 0.006 gm. MgO), and warm to 60°C. Add 1:1 ammonium hydroxide dropwise, with vigorous stirring, until a precipitate starts to form, then add dropwise an excess of 10 ml. of concentrated ammonia. Allow to settle for at least 20 minutes. Filter on a fritted Jena glass crucible (1-G-3) with suction. Transfer the precipitate with policing, and wash four times with cold 1:200 ammonium hydroxide. Discard the filtrate.

VOLUMETRIC METHOD

Dissolve the precipitate by means of 30 ml. of hot 1:3 hydrochloric acid, added in small quantities and drawn through the filter with suction; wash thoroughly with hot water. Collect the solution in a 500-ml. Erlenmeyer flask and dilute to about 100 ml. Cool the flask to about 20°C. Add 1 gm. of potassium bromide crystals and a few drops of methyl red and run in a standard potassium bromate solution from a burette until the pink color is destroyed. Allow to stand for about one minute to complete the bromination reaction. (A spot test may be made, using a drop of the solution, a drop of potassium iodide solution, and a drop of starch solution to determine whether the bromate is present in excess. The test should yield a blue color.) Add 5 ml. of fresh 20% potassium iodide solution (resulting in a brown color or brown precipitate) and titrate with standard thiosulfate until most of the brown color has disappeared. Add 1 ml. of starch solution, and continue the titration to the disappearance of blue color. Calculate the bromate equivalent of the thiosulfate, and subtract from the original bromate volume to find the net volume equivalent to the MgO. Calculate the percentage of MgO.

Net titration $\times$ MgO factor $\times 100 = \%$ MgO.

(*1*) *Standard Potassium Bromate* (*N/5*) *Solution:* Dissolve 120 gm. of potassium bromate in distilled water and make up to 20 liters. Standardize against a known magnesium chloride solution by precipitating the quinolate as in the regular procedure, filtering, washing, dissolving with hydrochloric acid, and titrating, using *N*/10 sodium thiosulfate for the back titration.

(*2*) *Sodium Thiosulfate* (*N/10*) *Solution:* The proper quantity of thiosulfate for preparing a liter of exactly 0.10*N* solution may be purchased in sealed glass tubes. It should be dissolved in freshly boiled and cooled distilled water and diluted in a volumetric flask. To obtain the equivalent of 1 ml. of thiosulfate in terms of standard bromate, add 10 ml. of bromate from a burette to 100 ml. of water containing 5 ml. of concentrated hydrochloric acid; then add 10 ml. of 20% potassium iodide solution, and titrate the liberated iodine with thiosulfate, using starch as indicator near the end point. The volume of bromate divided by the volume of thiosulfate (expressed to two decimal places) gives the volume of bromate equivalent to 1 ml. of thiosulfate.

(*3*) *Standard Magnesium Chloride Solution:* This may be prepared by dissolving 1 gm. of pure MgO in hydrochloric acid and diluting to 2 liters. The exact MgO concentration may be checked by a gravimetric determination (either as pyrophosphate or as quinolate ignited to MgO) on a 50-ml. aliquot.

(*4*) *8-Hydroxyquinoline Solution* (*5%*): To 5 gm. of 8-hydroxyquinoline add 12 ml. of glacial acetic acid. Stir until mostly dissolved and dilute with 75 ml. of distilled water. Heat to 60°C. and stir until solution is complete, then filter into an amber bottle and dilute to 100 ml.

(*5*) *Potassium Iodide Solution:* Dissolve 5 gm. of potassium iodide in 25 ml. of distilled water and filter (if necessary) into an amber bottle. The solution deteriorates on long standing and should be water-white when used.

(*6*) *Starch Indicator:* Mix 2 gm. of soluble starch with a little cold water and add to 100 ml. of boiling water. Cool and add 10 ml. of 25% potassium iodide and 10 ml. of 10% sodium hydroxide solutions. This solution keeps well over a fairly long period of time.

Gravimetric Procedure

The magnesium precipitate may be dried at 130° to 140°C. on the fritted glass filter and weighed if desired. It is also permissible to filter through paper and ignite the precipitate to MgO in a platinum crucible. Both procedures have given satisfactory results in the quantities ordinarily present in soda-lime glasses.

(2) Second Aliquot (Sodium Oxide)

Evaporate the 10-ml. aliquot in a 50-ml. beaker to about 1 ml. on a hot plate. Cool, and add slowly 20 ml. of the zinc uranyl acetate reagent with stirring. Allow the precipitate to settle for at least 20 minutes at room temperature, then filter on a weighed fritted glass filter (1-G-3) with suction. Transfer the precipitate completely by means of several small quantities of the precipitating solution contained in a wash bottle, using a rubber policeman. Wash the precipitate on the filter twice with 2-ml. quantities of the same solution, then six times with small volumes of 95% ethyl alcohol that has been saturated at room temperature with sodium zinc uranyl acetate. Allow the wash solution to drain through completely between washes. Finally, wash the precipitate twice with 5-ml. portions of anhydrous ether, added from a pipette. Continue to draw air through the filter for a minute or two after the precipitate appears dry, wipe off the crucible, set it aside near the balance for 10 or 15 minutes, and weigh. The empty crucible may be washed and dried in the same manner at the start and weighed as soon as it reaches room temperature.

$$\text{Wt. of precipitate} \times 20.15 = \%\ Na_2O.$$

Zinc Uranyl Acetate Reagent: Dissolve 100 gm. of uranyl acetate ($2H_2O$) in 490 ml. of water by warming on steam bath. Dissolve separately 300 gm. of zinc acetate ($3H_2O$) in 320 ml. of water, also with warming, and combine the two solutions. Add 90 ml. of 30% acetic acid solution. If the solution remains clear, add a few crystals of sodium chloride so as to saturate the solution with respect to sodium zinc uranyl acetate. Filter small amounts of the reagent as required.

(3) Third Aliquot (Potassium Oxide)

Evaporate the reserved 50-ml. aliquot to dryness in the 75-ml. platinum dish, and heat on hot plate to eliminate all perchloric acid. Heat the dish cautiously over a small flame, avoiding loss by spattering, until all of the perchlorates are decomposed. Finally, heat the dish briefly to a dull red. Cool, rinse down the sides of the dish with a little water, and add about 1 ml. of concentrated hydrochloric acid and 0.5 ml. of chloroplatinic acid solution. Evaporate nearly to dryness on steam bath so that the residue solidifies on cooling. Add about 20 ml. of ethyl alcohol of 80% strength, stir to dissolve all soluble salts, and filter through a fritted glass filter (1-G-3) with suction. Wash the residue once or twice by decantation, transfer to the filter, and wash two or three times more on the filter, using 80% ethyl alcohol. Using a 100-ml. beaker as the receiver, rinse out the dish with hot water, pour it through the filter, and wash thoroughly to dissolve all potassium chloroplatinate. Add 2 ml. of concentrated hydrochloric acid and a strip of magnesium ribbon to the solution and warm while stirring until all of the platinum is reduced. Add more magnesium if necessary, and finally boil the solution for a few minutes. The settled solution will be colorless if the reduction of the chloroplatinate is complete. Filter out the platinum and wash several times with hot water. Ignite in a porcelain crucible in a muffle, cool in a desiccator, and weigh.

$$\text{Wt. of platinum} \times 0.4825 \times 200 = \%\ K_2O.$$

Chloroplatinic Acid Solution: Dissolve 15 gm. of platinum chloride ($H_2PtCl_6 \cdot 6H_2O$) in 50 ml. of water containing 5 ml. of 1:1 hydrochloric acid (one ml. will contain about 0.1 gm. of platinum equivalent to 0.05 gm. of K_2O).

(C) Sulfate

Decompose 3 gm. of glass by hydrofluoric-perchloric acid treatment exactly as described under section III (B); transfer the solution to a 600-ml. beaker. Dilute

to about 200 ml., neutralize with ammonia, then add hydrochloric acid to about 2-ml. excess. Cover the beaker, heat to boiling, and add 10 ml. of 10% barium chloride solution dropwise from a pipette. Continue boiling for 10 minutes, transfer to steam bath, and digest for 45 minutes. Filter through a tight paper (S & S No. 589 blue ribbon), transfer the precipitate completely, and wash with 4 portions of warm water. Ignite slowly in a tared porcelain crucible until the carbon is burned off and finally heat at about 1000°C. for 10 minutes, cool in desiccator, and weigh.

$$\text{Wt. of } BaSO_4 \times 0.343 \times \frac{100}{3} = \%\ SO_3.$$

(D) Boron

Grind 1 gm. of powdered glass and 2 gm. of sodium carbonate intimately together in an agate mortar. Transfer to a platinum crucible and heat until a clear melt is obtained. Rotate the crucible while cooling so that the melt solidifies in a thin layer. Nearly fill the crucible with distilled water, cover, and allow to digest on a steam bath until the melt is softened. Remove, rinse the cover, and allow contents of the crucible to evaporate nearly to dryness. Cool the crucible, add a drop of 0.04% bromcresol purple indicator, then 1:1 hydrochloric acid drop by drop, with stirring, until the mixture is just acid. Add 1 ml. of concentrated hydrochloric acid in excess. Transfer to a 100-ml. glass-stoppered cylinder, rinse the crucible several times with small quantities of water, and dilute to 25 ml. Add 25 ml. of absolute ethyl alcohol and 50 ml. of anhydrous ether. Stopper the graduate, and mix the contents by vigorous intermittent shaking over a 5-minute period. Finally allow the cylinder to stand for an hour or more, or until the two liquid layers have separated completely. Note and record the volumes of the two layers. Pipette out 50 ml. of the upper layer (ether-alcohol), and transfer to a 200-ml. Erlenmeyer flask. Add 1 drop of bromcresol purple indicator, then a normal solution of sodium hydroxide dropwise (with thorough shaking) until a permanent blue end point is reached. Add 1 ml. of 0.5% phenolphthalein solution, and continue the addition of the sodium hydroxide to a pink end point. Finally add an excess amounting to about three times the volume used between the two end points. Add about 50 ml. of water, and warm the solution carefully on the steam bath until the ether layer has evaporated, then transfer the flask to a hot plate and allow to boil until most of the alcohol has been eliminated. Cool, add 1:1 hydrochloric acid drop by drop until the solution is just acid, then add normal sodium hydroxide to faint alkalinity. (With glasses containing zinc, this procedure results in its complete precipitation as the hydroxide, which may be eliminated by filtration. This step also removes all of the iron, which is apt to be troublesome if present in abnormal amounts.) The solution is next treated with $N/10$ hydrochloric acid until definitely acid (5 to 10 drops excess), warmed on the steam bath, attached to a vacuum line, and allowed to boil under reduced pressure for a few minutes to eliminate dissolved gases. Cool to room temperature, add $N/20$ sodium hydroxide from a burette until the color changes from yellow to blue, and record the burette reading. Add about 1 gm. of mannite, and continue the titration to a permanent pink color, which is not destroyed by further additions of mannite. The volume between the two end points, corrected by a blank determination on the reagents, provides a measure of the boric acid contained in the aliquot portion of the ether layer. It is then possible to calculate the amount of boric acid contained in the entire ether layer and, from a knowledge of the distribution ratio, the quantity present in the aqueous layer. The boric-oxide content of the sample is arrived at by the following calculation:

$$\text{Ml. } N/20\ NaOH \times \left(a + \frac{b}{K}\right) \times 0.00348 = \%\ B_2O_3.$$

Wherein a = vol. of ether layer (ml.).
b = vol. of aqueous layer (ml.).
K = partition coefficient.

Notes: The value of K (partition coefficient) is taken as 0.36 for ordinary laboratory temperatures of about 25°C. If the extraction is made at a room temperature of 30°C. or higher, the value becomes 0.34.

The blank determination is made conveniently by repeating the entire procedure on a glass (as Standard Sample No. 80) known to contain no boron.

IV. Analytical Results

Tables I, II, and III contain a record of results of analyses of Bureau of Standards glass samples by the methods described. The figures, which include results obtained by two different operators over a period of several months, are intended to show normal variations

TABLE I

RESULTS ON STANDARD GLASS NO. 128*

SiO_2	"R_2O_3"	CaO	MgO	BaO	Na_2O	K_2O	SO_3	B_2O_3
69.37	1.96 (*a*)	4.73	3.34 (*a*)	0.46	16.68	0.97	0.35	1.54
69.43	1.94 (*a*)	4.72	3.33 (*a*)	0.50	16.74	1.01	0.34	1.50
69.53	2.00 (*b*)	4.75	3.37 (*a*)	0.48	16.72	0.99	0.36	1.49
69.41	2.01 (*b*)	4.73	3.38 (*b*)	0.49	16.80	1.00	0.37	1.56
	2.00 (*b*)	4.77	3.41 (*b*)			1.00		
69.51†	1.96†	4.76†	3.33†	0.49†	16.83†	0.99†	0.35†	1.52†

* All results corrected to 0.18% ignition loss.
† Bureau of Standards recommended values.
(*a*) Volumetric. (*b*) Gravimetric.
Note: All silica separations were made by a single dehydration in perchloric acid.

rather than check values that might be obtained in duplicate analyses made by one analyst.

TABLE II

RESULTS ON STANDARD GLASS NO. 80*

SiO_2	"R_2O_3"	CaO	MgO	Na_2O	K_2O	SO_3
74.16	0.45 (*b*)	4.70	3.21 (*b*)	16.60	0.03	0.39
74.02	0.40 (*b*)	4.72	3.30 (*b*)	16.65	0.05	0.41
74.12	0.42 (*b*)	4.68	3.27 (*b*)	16.71	0.05	0.43
	0.44 (*b*)	4.75	3.27 (*b*)	16.66	0.04	0.41
74.09†	0.42†	4.65†	3.23†	16.65†	0.04†	0.41†

* All results corrected to 0.30% ignition loss.
† Bureau of Standards recommended values.
(*b*) Gravimetric.
Note: All silica separations were made by double dehydration in perchloric acid.

TABLE III

BORON DETERMINATIONS ON STANDARD GLASSES

Standard No. 92 (%)	Standard No. 93 (%)
0.75	12.74*
0.74	12.70*
0.77	
0.70†	12.76†

* Determined on 0.5-gm. sample.
† Bureau of Standards recommended values.

V. Discussion of Procedure

One important requirement in an analytical procedure involving hydrofluoric acid treatment is the elimination of all traces of fluorine. Residual fluorine, even in very small amounts, prevents the complete precipitation of alumina and results in a positive error in the magnesia, as was demonstrated several times in the preliminary work on these methods. The addition of boric acid at the end of the decomposition period seems to eliminate this difficulty.

A single dehydration with perchloric acid, following fusion of the glass with a small excess of sodium carbonate, has shown nearly complete recovery of silica in checks on Standard Glass No. 128. Making the fusion directly in the dish in which dehydration takes place eliminates any transfer of the sample. With certain glass compositions, an accurate silica determination may require a second dehydration, and this may be done readily by evaporating the small filtrate directly in a Pyrex-brand beaker, fuming briefly, diluting, filtering, and washing as before.

The analytical procedure in general is capable of a great many modifications. It may be desirable at times to use a separate sample or aliquot for each element to be determined, thus permitting a number of analysts to take part in the work. The use of such "unit" samples is quite possible in silicate analysis, provided certain points are kept in mind. The following observations apply to this type of analysis, assuming that one large sample or a number of small samples of a glass have been decomposed and silica and fluorine have been eliminated.

(1) Calcium oxalate may be precipitated in the presence of alumina either in faintly acid or in ammoniacal solution. In the latter case, alumina, if already precipitated with ammonium hydroxide, goes back into solution quantitatively on boiling with ammonium oxalate. Iron may accompany the calcium precipitate, but this is not a serious factor with flint glasses.

(2) Calcium may also be determined without prior removal of BaO, provided sufficient sulfuric acid is present in the solution to convert the barium to $BaSO_4$. (This statement applies, of course, only to volumetric determinations of CaO.)

(3) Magnesium may be precipitated as quinolate and titrated in the presence of calcium oxalate.[6] There is no objection to the presence of barium as precipitated $BaSO_4$. In making a direct MgO determination, then, it is necessary only to (*a*) precipitate and remove "R_2O_3" as hydroxides, (*b*) add a slight excess of sulfuric acid to combine with BaO, (*c*) precipitate CaO as oxalate, and (*d*) precipitate MgO as quinolate, filter out the combined precipitate, and titrate the MgO with standard bromate.

(4) Sodium, potassium, barium, and sulfate determinations are made directly on separate samples or aliquots as in the procedure outlined. Silica and boron are always determined on individual samples of the glass.

VI. Operating Schedules

Figures 1 to 4 are intended to show the approximate time required for completing a set of glass analyses according to several possible procedures. The time required for the various operations is shown roughly on the scale at the left, and in each case it is assumed that four different glasses are being handled at one time.

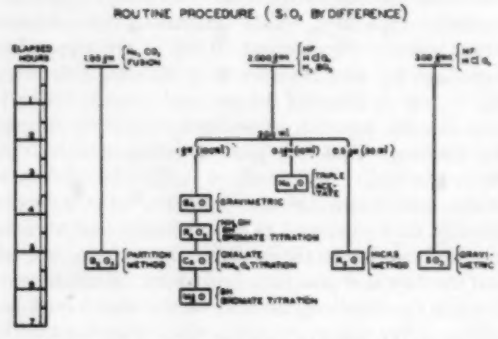

FIG. 1.—Plan No. 1 for glass analysis.

The scheme shown in Fig. 1 (plan No. 1) follows in general the procedure which has been described in detail, except that silica is taken by difference. If the glasses contain no boron or sulfate (or quantities that may be considered constant), the procedure is considerably simplified and is ideal for use in a one-man laboratory.

[6] J. C. Redmond, "Precipitation and Titration of Magnesium Oxyquinolate in Presence of Calcium Oxalate and Its Application in Analysis of Portland Cement and Similar Silicates," *Bur. Stand. Jour. Research*, **10** [6] 823–26 (1933); R.P. 569; *Ceram. Abs.*, **12** [9] 343 (1933).

Figure 2 outlines a procedure that would be particularly useful where several analysts are available for routine work. It provides for dividing a single

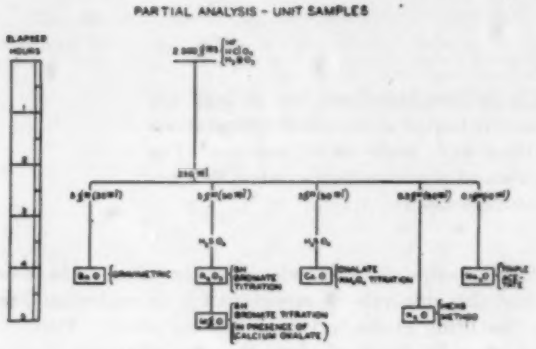

FIG. 2.—Plan No. 2 for glass analysis.

large sample into five aliquots and would make it possible to handle a large number of samples in one working day. As in plan No. 1, silica is taken by difference, and it is assumed in plan No. 2 that SO_3 and B_2O_3 need not be determined.

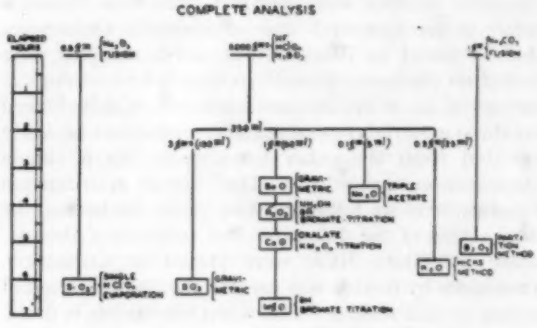

FIG. 3.—Plan No. 3 for glass analysis.

Figure 3 (plan No. 3) shows the possibility of decomposing a single large sample (5 gm.) and using aliquot portions for most of the constituents, including SO_3. Silica and boric oxide are determined as usual on separate samples, and it is possible to complete all operations in the space of one working day.

Figure 4 (plan No. 4) shows a possible procedure for a control analysis which might be of value for frequent checks on the glass from a continuous furnace. Here it is assumed that determinations of alumina, lime, and

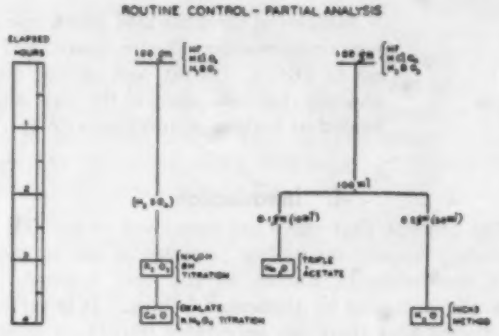

FIG. 4.—Plan No. 4 for glass analysis.

alkalis will give an adequate picture of composition changes. Barium (if present) is precipitated but not determined, sulfate and boric oxide are assumed to remain constant, and magnesia is calculated from the CaO content. It is indicated that a brief check of this kind could be completed in four hours.

VII. Summary

A procedure has been outlined which makes it possible for one analyst to determine all of the major constituents in a soda-lime glass in one working day with a good degree of accuracy. The methods may doubtless be applied to other silicates with some change in details, but until the accuracy can be established by check analyses of standard samples, the procedure should be confined to analysis of glass.

Acknowledgment

The authors wish to acknowledge the cooperation of G. E. F. Lundell and various members of the staff at the National Bureau of Standards, who assisted in the development and trial of the analytical procedures.

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INVESTIGATION OF THE EFFECT OF HEAT ON THE CLAY MINERALS ILLITE AND MONTMORILLONITE *

By R. E. Grim and W. F. Bradley†

Abstract

Samples of three purified illites, one purified montmorillonite, and one natural clay containing montmorillonite, quartz, and limonite were heated at successive temperatures up to 1400°C. X-ray and optical determinations were made on all samples. The changes that take place in the clay minerals, illite and montmorillonite, when they are heated at various temperatures up to 1400°C. are discussed.

I. Introduction

The concept that clays are composed essentially of extremely minute crystalline particles of one or more of a few minerals, known as the clay minerals, is generally accepted by students of clays. It is further recognized that there are only three important groups of clay minerals (Table I), that is, almost all clays are composed of extremely small particles (frequently less than 0.001 mm. in diameter) of one or more of these three clay mineral groups.

Table I

Important Clay Mineral Groups*

Name	Chemical composition	Occurrence
Kaolinite	$(OH)_8Al_4Si_4O_{10}$	China clay, fire clay, ball clay, etc.
Illite	$(OH)_4K_y(Al_4 \cdot Fe_4 \cdot Mg_4 \cdot Mg_6)(Si_{(8-y)} \cdot Al_y)O_{20}$	Shale, fire clay, ball clay, etc.
Montmorillonite	$(OH)_4Al_4Si_8O_{20} \cdot xH_2O$	Bentonite, gumbotil, loess, etc.

* For details concerning other clay minerals and possible variations in the composition and properties of clay minerals generally, see R. E. Grim, "Relation of Composition to Properties of Clays," *Jour. Amer. Ceram. Soc.*, 22 [5] 141–51 (1939).

Considerable study has been made of the changes that take place in kaolinite[1] when it is heated at various elevated temperatures, but no detailed study appears to have been made of the effect of heat on illite and montmorillonite. As montmorillonite and illite are the dominant constituents of many clays, and minor but important constituents of other clays used for ceramic purposes, a knowledge of the effect of heat on these clay minerals is important for an understanding of the firing characteristics of many clays. Further, the firing behavior of clays that are mixtures of clay minerals cannot well be understood until the firing characteristics of the pure clay minerals have been studied. It is the purpose of this report to give the results of an investigation of the changes that take place when illite and montmorillonite are heated at various temperatures up to 1400°C.

II. Procedure

Samples of illite were purified from two Pennsylvanian underclays and one argillaceous Ordovician dolomite found in Illinois. The crude samples were selected on the basis of previous detailed mineralogical analyses of many argillaceous materials, which showed that these materials contained illite that could be easily separated from the other constituents by a simple sedimentation procedure. The actual purification procedure was as follows: The clays (including the clay fraction of the dolomite after removal of the carbonate by dilute HCl) were placed in suspension. Ammonium hydroxide was used as the dispersing agent because no salt residue forms when the sample is dried. The suspension was allowed to stand until material coarser than 0.001 mm. had settled out. The suspension remaining was removed and dried on a steam bath. This material, composed of particles finer than 0.001 mm., was shown by X-ray diffraction analyses to contain only illite as the clay mineral. A small amount of quartz that could not be removed in the sedimentation process was also present. Before firing, the dried material was ground to pass a 100-mesh sieve.

It has previously been reported[2] that clay minerals belonging to the illite group may vary somewhat in their physical characteristics and their chemical composition, notably in the K_2O content. One illite sample (No. 1) is representative of illite with relatively high plastic and bonding properties, slightly lower K_2O content, and smaller ultimate particle size. The other illite samples (Nos. 2 and 3) are representatives of illite with relatively larger ultimate particle size and low plastic and bonding properties. The chemical

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† Petrographer and Associate Chemist, respectively, Illinois State Geological Survey.

[1] (*a*) T. N. McVay and C. L. Thompson, "X-Ray Investigation of Effect of Heat on China Clays," *Jour. Amer. Ceram. Soc.*, 11 [11] 829–41 (1928).

(*b*) R. A. Heindl and W. L. Pendergast, "Fire Clays, Some Fundamental Properties at Several Temperatures," *ibid.*, 13 [10] 725–50 (1930).

(*c*) J. F. Hyslop, "Chemistry of Fire Brick," *Jour. West Scot. Iron Steel Inst.*, 40 [Part 2] 23–28 (1932).

(*d*) L. Tscheischwili, W. Büssem, and W. Weyl, "Metakaolin," *Ber. deut. keram. Ges.*, 20 [6] 249–76 (1939); *Ceram. Abs.*, 18 [10] 279 (1939).

[2] (*a*) R. E. Grim, "Relation of Composition to Properties of Clays," *Jour. Amer. Ceram. Soc.*, 22 [5] 141–51 (1939).

(*b*) R. E. Grim and W. F. Bradley, "A Unique Clay from the Goose Lake, Ill., Area," *ibid.*, pp. 157–64 (1939).

composition and source of the purified illite are given in Table II.

TABLE II

CHEMICAL COMPOSITION AND SOURCE OF SAMPLES*

Sample (1), purified illite from Pennsylvanian underclay, Grundy County, Ill.; (2), purified illite from Pennsylvanian underclay, Vermilion County, Ill.; (3), purified illite from Ordovician Maquoketa clayey dolomite, Kane County, Ill.; (4), purified montmorillonite from Cretaceous bentonite, Wyo.; and (5), bentonite from Cretaceous, near Pontotoc, Miss.

	Sample No.				
	(1)	(2)	(3)	(4)	(5)
SiO_2	52.79	51.22	51.10	64.60	57.55
Al_2O_3	24.99	25.91	21.96	20.80	19.93
Fe_2O_3	4.68	4.59	6.38	3.05	6.35
FeO	1.10	1.70	1.65	0.46	0.95
MgO	2.70	2.84	3.91	2.30	3.92
CaO	0.09	0.16	1.73	0.53	1.94
Na_2O	0.20	0.17	0.03	2.60	0.33
K_2O	5.86	6.09	6.62	0.40	0.59
TiO_2		0.53	0.46	0.15	0.32
Ignition loss	7.14	7.49	6.24	4.74	8.53
Total	99.55	100.70	100.08	99.63	100.41
H_2O+	6.83	7.14	5.75	4.74	8.51
H_2O-	5.56	1.45	1.74	8.00	7.43

* Analyses made under the supervision of O. W. Rees' Chemist, Illinois State Geological Survey.

One sample of montmorillonite (No. 4) was purified from a sodium bentonite (pH = 9.04) by the same process used for the illite samples. The purified sample was composed of particles less than 0.001 mm. in diameter, and X-ray diffraction analysis showed the presence of montmorillonite and about 10% of quartz. The other montmorillonite sample (No. 5) was an impure calcium-hydrogen bentonite (pH = 4.10), containing quartz in grains up to 0.1 mm. and limonite in addition to the montmorillonite. This latter sample was not purified.

Small separate portions (about 0.2 gram) of each sample were heated for two hours in platinum crucibles in an electric furnace at 100°, 150°, 200°C., and so on at 50°C. intervals, up to 1100°C. Additional portions of each sample were heated in Alundum crucibles in a gas-fired furnace for two hours at 1200°, 1300°, and 1400°C. In order to determine whether a two-hour firing approached equilibrium conditions, small portions of each sample were fired for six hours at 450°, 600°, and 800°C. No significant changes were apparent for these longer heatings, but the gradual appearance of some features would suggest that true equilibrium may not strictly have been attained. Equilibrium, however, is not critical in these studies. All temperature measurements were made by thermocouples. The heated samples were studied with the petrographic microscope and by means of X-ray diffraction analysis.

III. Changes in Physical Characteristics

(1) Illite

The three samples of illite, before firing, were light gray to light gray-green in color. All of the samples showed the same changes in characteristics up to about 1100°C.; they became very light yellow-red at 250°C., and this color darkened slightly with increased firing temperatures up to about 900°C. At 900°C., the samples were red-brown and had a loose powdery character; at 950°C., they retained the same color but had formed aggregates that could be broken with slight pressure. All samples were well vitrified be 1100°C., and they were completely vitrified at 1200°C. Sample (1) at 1300°C. was brown, dense, and slightly bloated; at 1400°C., it was a steel-gray glass with many large bloat holes. Sample (2) at 1300°C. was a steel-black glass with many bloat holes which had disappeared at 1400°C. Sample (3) showed a higher degree of vitrification at 1100°C. than samples (1) and (2). At 1200°C., it was a dark brown glass with many small bloat holes which had disappeared at 1300° and 1400°C.

It is interesting to note that the illite sample (No. 1) with the high plastic and bonding properties and slightly lower K_2O content is the only one that remained bloated at 1400°C. Sample (3), which contained the largest amount of iron, also exhibited relatively greater vitrification at a lower temperature than the other illite samples.

(2) Montmorillonite

The original samples of montmorillonite were light yellow-gray (No. 4) and light yellow (No. 5). At about 250°C., both samples became distinctly more yellow-red, and this color became darker on heating to successively higher temperatures. Both samples at 900°C. were light brown and showed no vitrification. At 950°C., they had the same color but had formed small powdery, easily broken aggregates, thus showing the beginning of vitrification. At 1050°C., both samples were partially vitrified and slightly bloated; at 1200°C., vitrification was about complete. Sample (4) at 1300° and 1400°C. was brown glass with many large bloat holes. Sample (5) at 1300° and 1400°C. was steel-gray glass, not bloated.

IV. Change in Mineral Composition of Illite on Firing

(1) Interpretation of Optical Data

The indices of refraction for the three samples of illite, plotted in Figs. 1, 2, and 3, show such marked general similarity that it may be concluded that about the same changes took place in all samples of this clay mineral. There was little change in the general appearance of all three samples as observed under the microscope on heating up to about 800°C., and it was possible to measure γ and α values on all samples heated to this temperature. The birefringence, shown by the difference between the γ and α values, was retained in samples fired at 700°C. and then began to decrease slightly in samples fired at higher temperatures. Samples fired above 800°C. were unlike those fired at lower temperatures. Samples fired between 800° and 1000°C. had the appearance of a heterogeneous aggregation of unlike material, and it was possible only to approximate the spread of the indices of refraction.

The retention of the birefringence and of the original appearance to a temperature of 800°C. would indicate that the illite structure was not completely lost before this temperature was reached. The reduction in the birefringence at 700°C. suggests that the final destruction of the illite lattice begins at about this temperature.

Sample (1), the relatively low K_2O illite with high plastic and bonding properties, unlike samples (2) and (3), shows an initial increase in indices of refraction on firing up to 200°C. The explanation for this increase is not entirely clear, but it probably results from the finer ultimate particle size of this illite as compared to the other illite samples with the attendant greater difficulty in removing adsorbed water.

From 350° or 400° to 600°C., there is a decrease in indices of refraction corresponding to the loss of (OH) water from the lattice.[3] Between 600° and 700°C., there is little change in the indices of refraction or in the birefringence, suggesting a period of existence of the illite from which (OH) water has been completely lost prior to its final breakup.

[3] U. Hofmann and J. Endell, "Dependence of Base-Exchange and Swelling of Montmorillonite on Preheating," *Z. Ver. deut. Chem.*, Beihefte No. 35, 10 pp. (1939).

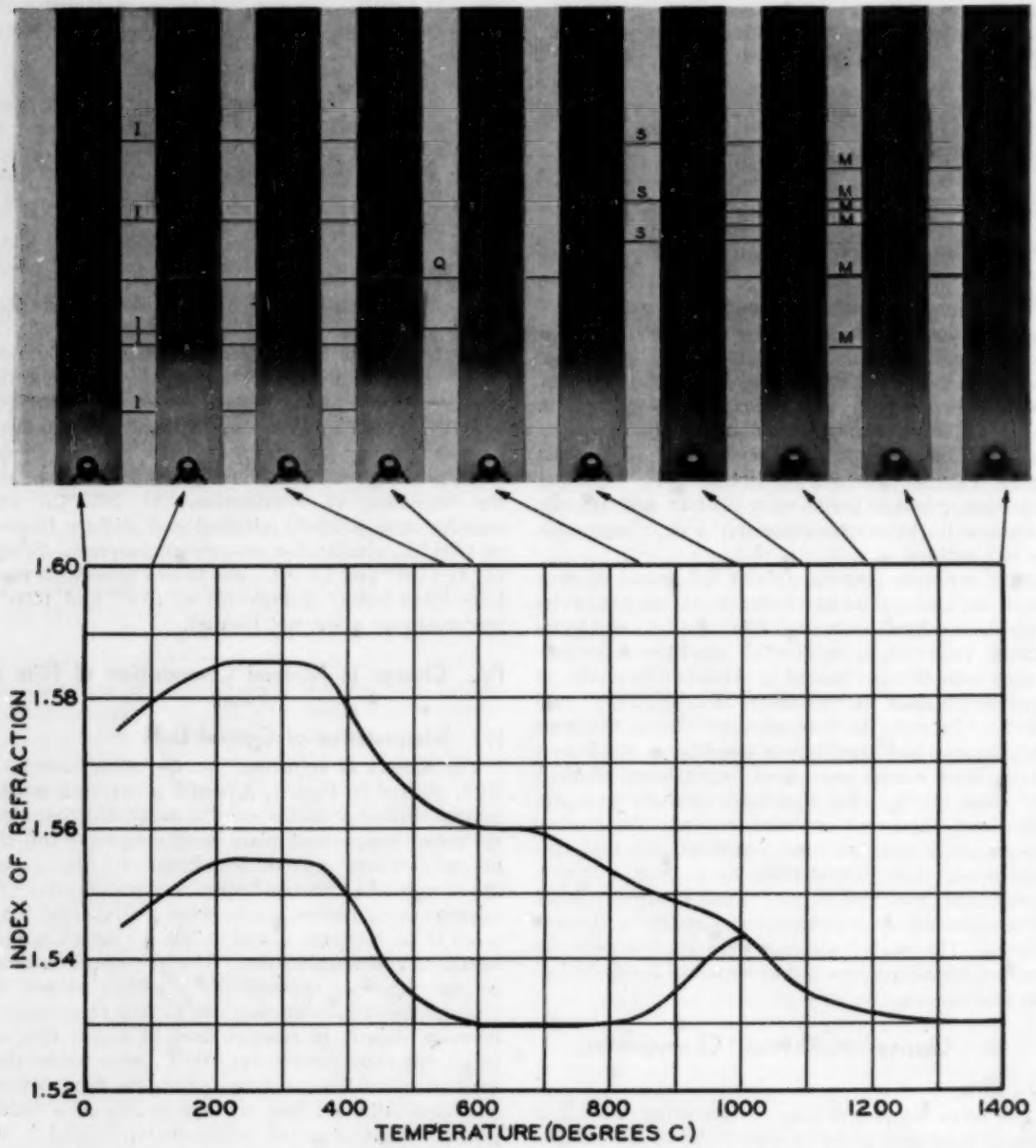

FIG. 1.—Indices of refraction and representative X-ray diffraction patterns of sample (1). The more prominent lines of various phases are designated as follows: I = illite, Q = quartz, S = spinel, and M = mullite.

The optical data gave no indication of the composition of the material heated to 800° to 1100°C. other than to indicate a heterogeneous material that is changing continually with increasing temperatures.

Samples heated to 1100°C. show the presence of glass (the indices given on Figs. 1, 2, and 3 for samples heated to 1100°C. and higher are for the glass component) through which are scattered many minute discrete particles that cannot be identified. Samples heated to 1300° and 1400°C. show needles of mullite disseminated through the glass.

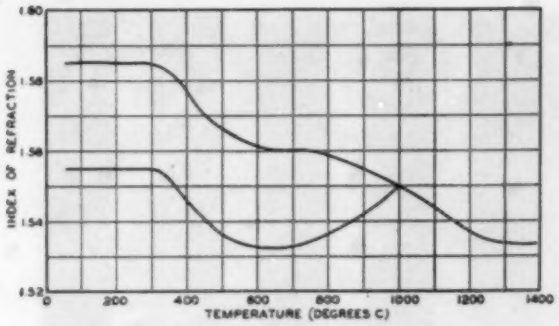

FIG. 2.—Indices of refraction of sample (2).

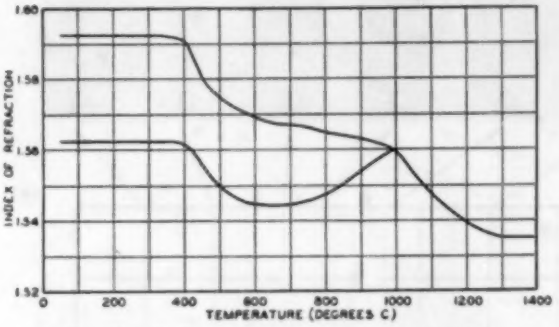

FIG. 3.—Indices of refraction of sample (3).

(2) Interpretation of X-Ray Data*

As might be inferred from the foregoing optical examination, the three illite specimens investigated exhibited parallel successions of phase changes, differing only in the apparent relative amounts and, to a small degree, in the temperatures at which new phases developed. Diffraction diagrams illustrating these changes are reproduced only for sample (1). The clay mineral persists in effect to about 850°C. in each sample. In agreement with the index curves, there are characteristic differences in the diffraction diagram before and after loss of (OH) water from the lattice. The positions of the 001 reflections are shifted slightly corresponding to an increase of 1 or 2% in the cell height, and the relative intensity of the 004 and 008 lines is noticeably greater for the dehydrated state. Above 600° to 700°C., the illite diffraction lines gradually disappear, being observed for the last time at 850°C. Small amounts of α-Fe_2O_3 have become apparent at 850°C., particularly in sample (3), but it seems probable that this iron constituted a limonite impurity in the original clays. The basic portion (MgO, Fe_2O_3, Al_2O_3) of the clay mineral gives rise, in this same temperature range, to the appearance of an entirely new phase, a spinel. The spinels comprise a highly variable group of cubic double (or mixed) oxides which have been the subject of extensive research.[4] The spinel developed in these three cases has a unit cube edge of about 8.15 Å and probably represents a composition (Mg.-Fe)(Al_2·Fe_2)O_4. Both the amount present and the particle size of the phase increase with increased ignition temperature through 1100° or 1200°C. By 1300°C., however, the spinel in each case has dissolved in the glass. In each sample, mullite needles appear in varying amounts, first becoming apparent at around 1100°C. and persisting beyond the solution of the spinel. The amounts of spinel and of mullite appear to be complementary, and the richer spinel specimens are subject to fusion at the lower temperatures. A minor amount of quartz, from which it was impractical to completely free the illite samples examined, persisted without any inversion to cristobalite until its gradual solution between 850° and 1050°C.

These spinel and mullite phases are of particular interest in comparison with the changes on firing of the crystalline micas. Muscovite[5] ignites to leucite and γ-Al_2O_3 and finally to α-Al_2O_3. A biotite which was examined yielded leucite, γ-Fe_2O_3, and a very little of a spinel similar to those mentioned before, around 1200°C., with γ-Fe_2O_3 remaining as a crystalline phase at 1350°C. These γ-oxide phases of course, are, themselves spinel-like phases, but they have different characteristic cube-edge lengths.

(3) Summary of Mineral Changes in Illite

Illite loses (OH) water from its lattice between about 350° and 600°C., retaining its essential micaceous character but assuming the form of a new "anhydrous modification." The final destruction of the illite lattice becomes complete around 800° to 850°C. The optical data suggest that the dehydrated lattice persists with little change between 600° and 700°C. and that following 700°C., the final destruction is gradual. The "anhydrous modification" exhibits a slightly larger unit cell height and distinctly lower refractive indices than does the normal mineral with its (OH) groups intact.

At 850°C., a spinel, a new phase, appears, which increases in size of particle and in amount with temperatures up to 1100° or 1200°C. The middle sheet of

* X-ray diffraction diagrams were obtained in the laboratories of the Chemistry Department of the University of Illinois through the courtesy of G. L. Clark. All diffraction diagrams were registered with FeKα radiations in a camera of 6.33 cm. radius (reduced 25%).

[4] T. F. W. Barth and E. Posnjak, "Spinel Structures with and without Variate Atom Equipoints," *Z. Krist.*, **82**, 325–41 (1932).

[5] A. Swetsch, "X-Ray Experiments in Ceramics," *Ber. deut. keram. Ges.*, **15**, 2–14 (1934).

the illite lattice, carrying the alumina, magnesia, and iron, seems to go into the formation of the spinel, and the alkalis and the silica from the outer sheets go into amorphous glass.

By 1300°C., the spinel has dissolved in the glass. Mullite becomes apparent at 1100°C. and persists to the final firing temperature employed in this work (1400°C.). A vitrified appearance at 950°C. suggests that glass formation has started at this temperature. The quartz present persists, without any inversion to cristobalite, until it is dissolved in the glass at about 1050°C.

V. Changes in Mineral Composition of Montmorillonite on Firing

(1) Interpretation of Optical Data

All of the fired samples of purified montmorillonite (No. 4), up to and including that fired at 800°C., retain the same appearance under the microscope and permit the determination of γ and α values (Fig. 4). The birefringence does not change at firing temperatures up to 600°C., but it gradually decreases as the firing temperature increases to 800°C. Samples heated above 800°C. show a rapid drop in the spread of the

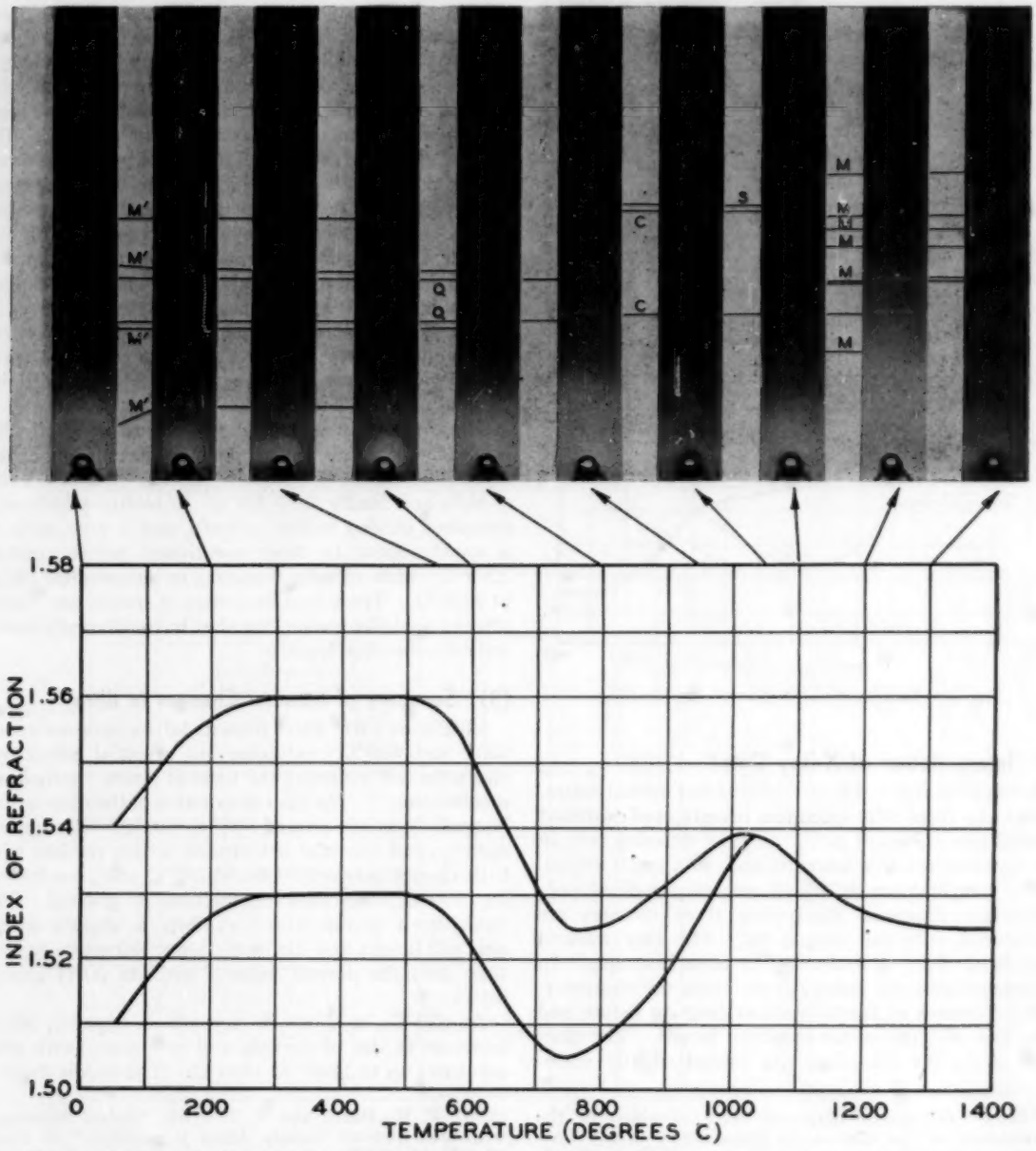

FIG. 4.—Indices of refraction and representative X-ray diffraction patterns of sample (4). The more prominent lines of various phases are designated as follows: M' = montmorillonite, Q = quartz, C = cristobalite, S = spinel, and M = mullite.

indices of refraction and a changed appearance under the microscope. It is suggested from these data that the final destruction of the montmorillonite lattice begins at about 600°C. and is essentially complete at about 800°C.

The indices of refraction increase as the temperatures of firing increase up to about 300°C. As for illite sample (1), it is suggested that this results from the slow loss of adsorbed water because of the very small ultimate particle size of the clay mineral. There is no change in the birefringence or decrease in the indices of refraction at firing temperatures up to 500°C. Above 500°C., the indices of refraction decrease, and above 600°C., the birefringence also decreases. This suggests that the (OH) water of the lattice begins to come off by 500°C. and is followed by the final destruction of the lattice. Like illite, an "anhydrous modification" develops with the loss of (OH) water from the lattice, but the temperature interval in which it is stable is much shorter than that for illite.

Only the range of indices of refraction can be measured on samples heated to temperatures between 800° and 1050°C. The components existing in this temperature range cannot be identified optically, and the

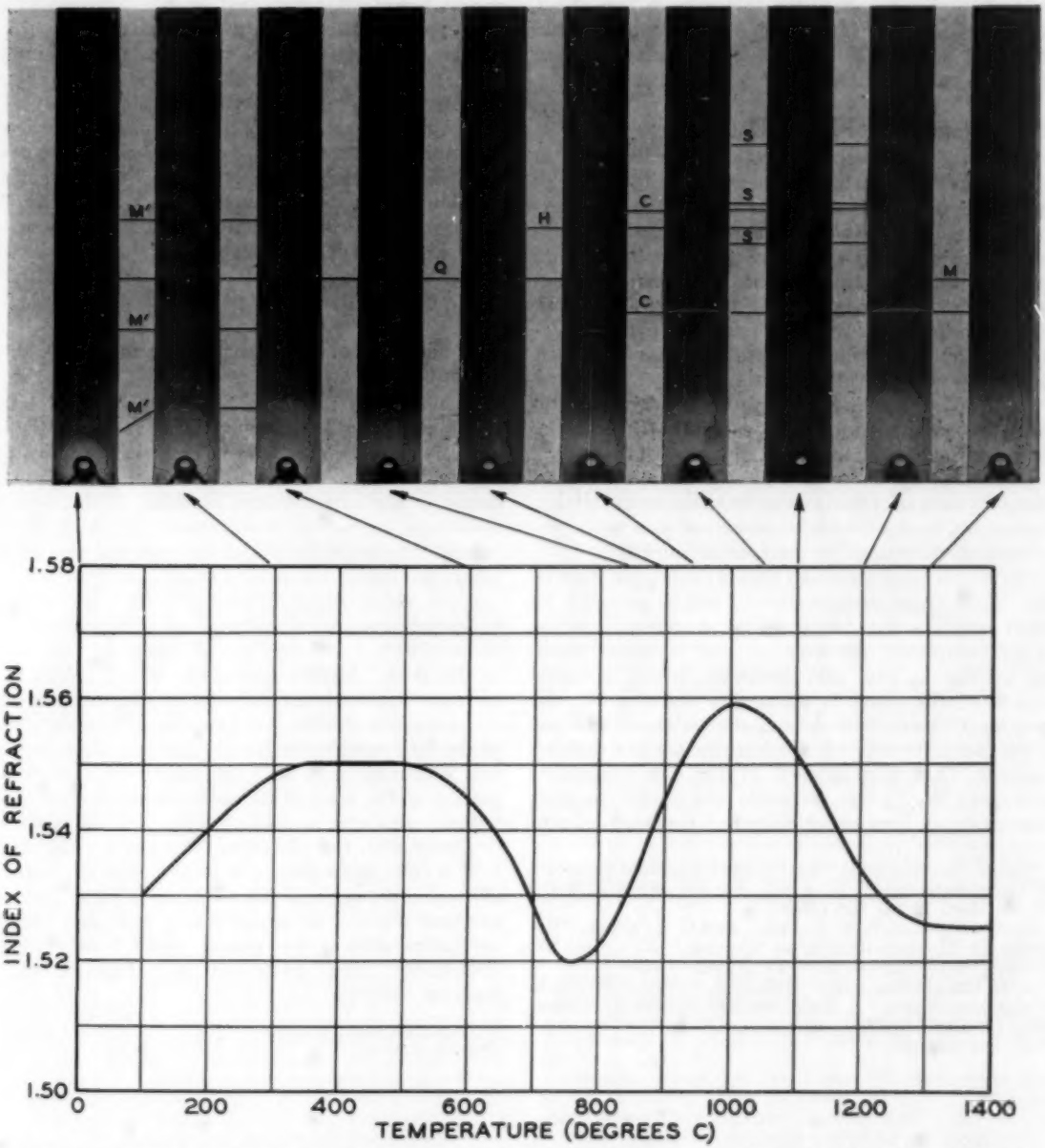

FIG. 5.—Indices of refraction and representative X-ray diffraction patterns of sample (5). The prominent lines of various phases are designated as follows: M' = montmorillonite, Q = quartz, H = hematite, C = cristobalite, S = spinel, and M = mullite.

optical data merely suggest that a heterogeneous changing mixture of components exists.

The sample fired at 1050°C. showed glass (the indices of samples fired to temperatures above 1050°C. on Fig. 4 are those of the glass) through which many discrete unidentified particles are scattered. Samples fired to higher temperatures showed successively more glass, and samples fired at 1300° and 1400°C. were largely glass with scattered needles of mullite.

Only a mean index of refraction could be measured for the fired samples of the impure montmorillonite, sample (5), and these data are given in Fig. 5. These optical data are too limited to be very significant, but the fact that the values for this sample run about parallel with those of sample (4) suggests that the changes in sample (5) were about the same as those that took place in sample (4).

(2) Interpretation of X-Ray Data

The two montmorillonite specimens investigated differed more from each other than did the illite samples among themselves, but, on the whole, they also exhibited similar successions of phase changes. Numerous investigations[8] of the behavior of montmorillonite toward low-temperature water have been reported and no attention has been given to this problem here. Interest is directed only to the water-free pyrophyllite-like product from which hydration water has been irreversibly lost. This material is first observed around 200° to 300°C. as a triple layer structure quite analogous to the micas, and, as in the case of the illites, an increase in the unit-cell height of about 1% is observable by about 600°C., which it is assumed relates to the loss of (OH) water from the actual lattice. This phase, again, might be spoken of as a new "anhydrous modification" of the normal mineral.

The rather pure specimen (No. 4) exhibited lines of this "anhydrous modification," which persisted to about 800°C. By 850°C., the montmorillonite is rather completely decomposed, and a spinel phase has appeared. For this specimen, which contains little iron, the phase is apparently spinel itself, the cube length being 8.05 Å, and the estimated amount of the phase developed is the least for the five samples studied. The crystallization of the raw unpurified bentonite (No. 5) was distinctly less perfect to start with, and clay lines easily discerned persisted only to about 600°C. Little more can be observed until around 850° to 900°C., where both α-Fe_2O_3 and a spinel phase become apparent. The α-Fe_2O_3 appears first, suggesting that it develops from the limonite impurity known to be present rather than from the clay minerals. Between 1050° and 1200°C., the amount of α-Fe_2O_3 decreases, whereas the cube-edge length of the spinel grows from about 8.1 to 8.2 Å, evidently by dissolution of the α-Fe_2O_3.

The spinels in each case grow and become more abundant and then fuse into the glass, just as in the case of the illites. Mullite appears, as before, around 1050° or 1100°C., and the mullite-spinel proportions appear complementary; that is, No. 4 develops very little spinel and abundant mullite, whereas No. 5 develops abundant spinel and only a few mullite needles. Each of the montmorillonite samples precipitates considerable crystalline silica at about 950°C. In the case of sample No. 4, contaminated with very fine-grained quartz, it is not certain whether the newly synthesized silica is quartz or cristobalite, for inversion is taking place in the same range. In sample No. 5, however, it is distinctly apparent that quartz precipitates as such and that it later inverts to cristobalite. In each case, the cristobalite fuses with the glass at about the same temperature as the spinel.

(3) Summary of Mineral Changes in Montmorillonite

Final destruction of the montmorillonite lattice begins at about 600°C., immediately following the loss of (OH) lattice water, and is essentially complete at 800° to 850°C. The loss of (OH) water is first reflected in the optical values at about 500°C. by a reduction in the indices of refraction. A slight increase (about 1%) in the height of the unit cell also accompanies the loss of the (OH) water.

A new phase, spinel, appears at 850°C. and increases in particle size and abundance up to temperatures of about 1100°C. By 1300°C., all the spinel is dissolved in the glass. Mullite appears at about 1050°C. and increases in abundance with higher firing temperatures. As in the case of illite, it appears that the middle sheet of the montmorillonite lattice, carrying alumina, iron, and magnesia, goes to form spinel. A considerable portion of the silica of the outer sheets, however, contributes synthetic crystalline silica, and the remainder combines with the alkalis to form glass. The formation of amorphous glass, the development of crystalline silica, and the beginning of vitrification are first evident at about 950°C. In sample No. 5, it is clear that the crystalline silica is first quartz, which later inverts to cristobalite. The cristobalite is dissolved in the glass by 1300°C.

Illinois Geological Survey
Urbana, Illinois

[8] (*a*) G. Nagelschmidt, "Lattice Shrinkage and Structure of Montmorillonite," *Z. Krist.*, **93** [6] 481–87 (1936); *Ceram. Abs.*, **15** [8] 258 (1936).
(*b*) W. F. Bradley, R. E. Grim, and G. L. Clark, "Behavior of Montmorillonite on Wetting," *Z. Krist.*, **97**, 216–22 (1937); *Ceram. Abs.*, **17** [8] 288 (1938).
(*c*) E. Maegdefrau and U. Hofmann, "Crystal Structure of Montmorillonite," *Z. Krist.*, **98**, 299–323 (1937); *Ceram. Abs.*, **18** [2] 58 (1939).
(*d*) See footnote 3.

CERAMIC ABSTRACTS

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Ross C. Purdy, Editor

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Journal Reference: The journal name appears in italics, followed by the volume (bold-faced), issue number (in brackets), page numbers, and year (in parentheses).

August, 1940
19—8

Abrasives

Effect of sesquioxides on the process of formation of silicon carbide. M. V. Kamentsev. *Zhur. Priklad. Khim.*, **12**, 1330–37 (1939).—The investigation was conducted with a small Acheson-type 4-kw. furnace and a graphite crucible in a Tammann furnace. The batch was formed of industrial materials of high purity, to which was added up to 6% of aluminum and iron oxides. Contrary to the prevailing opinion, K. found that iron oxides in amounts up to 2 to 3% have a positive effect on the formation of silicon carbide, increasing its output and the dimensions of the crystals. Alumina has a negative influence on the process, inhibiting the formation of silicon carbide and leading to the formation of small poorly developed crystals. P.B. & E.S.

Superfinish. D. A. Wallace. *S. A. E. Jour.*, **46**, 69–92 (1940).—Methods of surface finish are reviewed and compared. Superfinish technique produces surfaces differing from all other types of machined surface finishes. It includes the following features: (1) use of bonded abrasive tools with firmly embedded coarse grits, so that only the cutting points are exposed; these are made to fit the work and can be large or small and curved or any shape necessary; (2) multimotion of the abrasive tool by oscillation of the abrasive block and traversing of the holder, besides the rotation of the work; (3) abrasive pressure measured in pounds; after the superfinish is produced, the stone is glazed and loaded and no further cutting action takes place; the next part has a rough surface, which dresses the stone again; (4) lower abrasive speed than other methods; (5) use of a lubricant rather than a coolant as there is not enough speed and pressure to make excessive heat; at the end of the cycle this lubricant carries the load and there is no more cutting; and (6) a short finishing time; the longest operation is 50 sec. Electron diffraction studies show that superfinishing produces an entirely new kind of surface, although only about 0.0001 to 0.0002 in. is removed. The relation of surface finish to lubrication is discussed thoroughly. In one test, a finely ground surface wore 46.5 mgm. off of a bearing; the superfinish surface wore off only 1 mgm. W.D.F.

Use of diamond-impregnated cemented carbide for core bits. W. C. Weslow. *Amer. Inst. Mining Met. Engrs. Tech. Pub.*, No. 1172; *Mining Tech.*, **4** [2] 12 pp. (1940).—Laboratory and field work have shown that (1) tungsten carbide and cobalt are the most desirable as a matrix as they are hardest; (2) smaller and less expensive bort diamonds can be used; (3) in exploratory drilling, such drill crowns are cheaper than hand-set crowns; (4) in blast hole drilling in harder formations, this may be much faster when a suitable drilling rig is available; (5) a rotary bit is a preventive measure against silicosis. When the rock is not hard enough to wear away the matrix and expose more diamonds, a silicon carbide sandblast may be used for resharpening. Field tests at the Soudan mine of the Oliver Iron Mining Co. in hard iron ore and jasper showed improved drilling speeds of 100 to 400% compared with those of jack bits. Best results were obtained at about 1000 r.p.m. and 1700 lb. total pressure. When drilling in an abrasive sandstone at the Noranda mine, no resharpening was necessary and the bit life was 1000+ ft. Advantages of the use of these bits are (1) no resetting, (2) 75% or more utilization of diamonds, (3) more footage per man-hour, (4) reduction in total rock to be drilled and in starting size of hole, (5) deeper holes without loss of gauge, and (6) less noise, vibration, and dust. W.D.F.

PATENTS

Abrading. C. H. Kasch (H. C. Simmons). U. S. 2,203,530, June 4, 1940 (Dec. 29, 1938).—A machine for grinding scalloped bevel edges.

Abrading machine. H. M. Zschau (Wodack Electric Tool Corp.). U. S. 2,201,420, May 21, 1940 (Sept. 13, 1937).

Abrasive article. N. P. Robie (Carborundum Co.). U. S. 2,205,276, June 18, 1940 (Feb. 11, 1937).—An abrasive article comprises abrasive grains and a bond comprising a partial acetal of a polymerized vinyl ester.

Abrasive composition. H. P. Nelson. U. S. 2,201,700, May 21, 1940 (Aug. 20, 1935; Jan. 18, 1939).—An abrasive composition includes particles of abrasive material

and a binder therefor comprising the reaction product of a magnesium oxychloride cement and zinc sulfide.

Adjustable water box for wet abrasive cutting machines. H. G. Robinson (American Chain & Cable Co., Inc.). U. S. 2,201,408, May 21, 1940 (March 3, 1938).

Apparatus for forming or dressing grinding wheels for use in form-grinding operations. Coventry Gauge & Tool Co., Ltd., J. E. Wainwright, and S. J. Harley. Brit. 521,001, May 22, 1940 (Nov. 8, 1938).

Apparatus for grinding double-edged razor blades. Christian Weidinger. Ger. 685,653, Nov. 30, 1939 (June 28, 1937); XII/67*c*. 5.—Method and apparatus are described. D.A.B.

Bonding adhesive for an abrasive article such as sandpaper. D. O. Guth (Minnesota Mining & Mfg. Co.). U. S. 2,202,765, May 28, 1940 (Sept. 11, 1930).

Boron carbide composition. J. A. Boyer and C. G. Rose (Carborundum Co.). U. S. 2,201,151, May 21, 1940 (Feb. 18, 1938).—A fused carbide composition consists essentially of boron carbide and a difficultly fusible carbide of the group consisting of tungsten, molybdenum, and chromium carbides in which the difficultly fusible carbide is present as a separate crystalline phase which has solidified from a fused mass.

Buffing machine. Herman Weber and J. R. De Hamer (Shakespeare Products Co.). U. S. 2,203,003, June 4, 1940 (Aug. 5, 1939).

Feeding mechanism for grinding wheel truing apparatus. W. H. Wood (Norton Co.). U. S. 2,203,608, June 4, 1940 (May 4, 1938; Jan. 4, 1939).

Floor-dressing machine. J. A. Gartner. U. S. 2,204,178, June 11, 1940 (Jan. 25, 1938).

Granular-coated material. R. L. Melton, R. C. Benner, and H. P. Kirchner (Carborundum Co.). U. S. 2,201,195, May 21, 1940 (Sept. 22, 1939).

Grinding apparatus. S. A. Hamnett. U. S. 2,203,262, June 4, 1940 (Aug. 29, 1938).—A tool-grinding apparatus.

Grinding machine. Jacob Decker (Cincinnati Grinders, Inc.). U. S. 2,205,373, June 18, 1940 (Nov. 7, 1938). C. G. Flygare (Norton Co.). U. S. 2,201,954, May 21, 1940 (Oct. 15, 1938). H. E. Kitchen (Farrel-Birmingham Co., Inc.). U. S. 2,202,587, May 28, 1940 (March 31, 1939).

Grinding tool. Hermann Schneider (Julius Pintsch K.-G.). U. S. 2,204,826, June 18, 1940 (May 26, 1939).—A grinding tool consists of an aluminum carrier and grains of grinding material pressed into the carrier, the tips of all the grains of grinding material standing out freely above the surface of the carrier and being located in an imaginary surface which is equidistant from the carrier surface and is complementary in form to the surface to be ground by the tool, and the embedding of the aluminum carrier comprising a layer of oxide consolidating the embedding of the grains of grinding material.

Grinding wheel. H. R. Simonds (Simonds Worden White Co.). U. S. 2,201,410, May 21, 1940 (Feb. 15, 1939).—In a grinding wheel, a disk-like rotatable backing plate having means for rigidly securing it to a driving element.

Hard carbide composition. J. A. Boyer and C. G. Rose (Carborundum Co.). U. S. 2,201,150, May 21, 1940 (Feb. 18, 1938).—A fused carbide composition consists essentially of boron carbide and a difficultly fusible carbide of the group consisting of vanadium, columbium, and tantalum carbides in which the difficultly fusible carbide is present as a separate crystalline phase which has solidified from a fused mass.

Honing tool. M. N. Prange (Detroit Hone Equipment Corp.). U. S. 2,201,593, May 21, 1940 (Feb. 28, 1938).

Indexing device for machines for grinding gear wheels. H. Birkbeck (Soc. Française d'Engrenages à Denture Rectifee). Brit. 521,062, May 22, 1940 (Nov. 12, 1938).

Knife-sharpening device. C. L. Jenks. U. S. 2,203,788, June 11, 1940 (April 1, 1937).

Machine for grinding both sides of knife blades. Ernst Schlächter. Ger. 682,005, Sept. 14, 1939 (Nov. 24, 1937); XII/67*a*. 6.—The abrasive stones are moved back and forth along the knife blade, one on each side of the blade. The knife blade is moved in the plane of the blade along a circulatory path, and, by a tilting or pushing of the blade transverse to its lengthwise axis, it comes into contact alternately with one grindstone and then with the other. D.A.B.

Machine for grinding the crankpins of crankshafts. Carl Mayer (Rudolf Nolle). U. S. 2,202,111, May 28, 1940 (Nov. 23, 1938).

Machine for grinding noncircular work. H. E. Balsiger and C. L. Ott (Landis Tool Co.). U. S. 2,201,218, May 21, 1940 (Oct. 22, 1935).

Machine for polishing rollers, such as used in printing machines. I. H. Gilbertson. U. S. 2,203,567, June 4, 1940 (May 9, 1938).

Making abrasive castings. Walter Siegert (Howard Foundry Co.). U. S. 2,204,453, June 11, 1940 (July 30, 1938).

Manufacture of abrasive articles. N. P. Robie (Carborundum Co.). U. S. 2,201,321, May 21, 1940 (April 27, 1936).

Manufacture of granular-coated materials. J. A. Williamson (Carborundum Co.). U. S. 2,201,196, May 21, 1940 (June 27, 1939).

Method and apparatus for manufacturing granular-coated webs. R. L. Melton, R. C. Benner, and H. P. Kirchner (Carborundum Co.). U. S. 2,201,194, May 21, 1940 (Sept. 23, 1933).

Method and means for producing or finishing gear teeth by a grinding or honing operation. Coventry Gauge & Tool Co., Ltd., J. E. Wainwright, and S. J. Harley. Brit. 520,557, May 8, 1940 (Nov. 8, 1938).

Motor-driven headstock for grinding machines, etc., and for other machine tools. A. W. Wigglesworth. Brit. 520,495, May 8, 1940 (Oct. 24, 1938).

Polishing and buffing machine. G. A. Carlson. U. S. 2,203,488, June 4, 1940 (June 30, 1938).

Producing porous abrasive objects. Deutsche Norton Ges. m. b. H. Ger. 686,017, Dec. 17, 1939 (Sept. 28, 1936); VI/80*b*. 11.—A grinding disk or wheel is made of ceramically bonded abrasive particles oxidizable at high temperatures, such as silicon carbide, boron carbide, or diamond. To the crude mixture of abrasive particles and ceramic binding agent, a readily oxidizing material at firing temperature such as aluminum, silicon, magnesium, boron, boron carbide, boron suboxide, or a mixture of these materials is added in such amount that the entire amount of oxygen present in the pores of the object and entering into the pores is compounded chemically with the pores upon firing under oxidizing conditions. D.A.B.

Pulpstone. C. L. Leafe (Norton Co.). U. S. 2,201,734, May 21, 1940 (June 9, 1938).

Relief grinding cutter blades. Ernest Wildhaber (Gleason Works). U. S. 2,204,714, June 18, 1940 (May 20, 1935; Oct. 29, 1937).

Surfacing machine abrasive sheet holder. E. B. George. U. S. 2,204,486, June 11, 1940 (Sept. 9, 1939).

Twist drill grinding attachment. J. A. Yantiss. U. S. 2,202,819, June 4, 1940 (May 2, 1938).

Valve seat grinding implement. S. H. Haas. U. S. 2,203,142, June 4, 1940 (Feb. 23, 1939). U. S. 2,203,143, June 4, 1940 (June 9, 1939).

Wheel dressing apparatus for form-grinding operations. Coventry Gauge & Tool Co., Ltd., and S. J. Harley. Brit. 521,022, May 22, 1940 (Nov. 10, 1938).

Art and Archeology

Buddhist baroque in Kashmir. C. Fabri. *Asia*, **39**, 593–98 (1939).—Terra-cotta heads in a museum at Lahore were traced back to the small village of Pambarvan where numerous badly broken pieces were found in the fields. Pieces from the same school were found at a monastery at Ushkar, built between A.D. 700 and 736, and at another

monastery at Harvan. The pieces show a Hellenistic influence, and the best were made by one of India's greatest sculptors. W.D.F.

Design in daily life. RECO CAPEY. *Jour. Roy. Soc. Arts*, **88** [4553] 343–63 (1940).—C. calls attention to design as influencing every activity in life, hence as drawing on all the possibilities in material and processes of manufacture and involving all the complex operations of the mind in the creation of an object. In substantiating this sphere of influence for design, R. reviews its prehistoric and historic development. Glass industries which adopted new techniques show profound effect in altering their decorative treatments, sandblasting and acid etching opening up a huge range of possibilities. Pottery is still pressed and thrown; most of the power is now electric, but the hand is still used in finishing and decorating. Transfer printing and transfer lithography, both comparatively new processes, may be confidently expected to develop new designs in pottery decoration. Advanced pottery design at present is restricted to surface expression. Cloisonné, although now made by machine, shows extremely good technique at a reasonable cost but lacks individuality of expression. R. comments: "In every craft the aspect of design changes not mainly because of fashion, but because new conditions bring with them new demands. Meanwhile, old masterpieces are never out of date." Illustrated. K.R.

Fresh finds of Mogul times. C. L. FABRI. *Asia*, **40**, 159–64 (1940).—This excavation was at Surkhanvali Ahli near the village of Divanpura in the Punjab. The period covered by this mound was from about A.D. 1400 to 1750. The importance of this excavation is that it shows the life of the common people which was not previously known, although there are extensive descriptions of the life of the upper classes. The site of the excavation was a bazaar of a provincial village. The houses are of pisé, or mud brick mixed with older fired brick. The rooms are small and mostly kitchens; some contain six hearths with eighty pottery vessels. There are large vessels for cooking rice, plates, platters, saucers, etc., and a spice-grinding stone and pestle. Some vessels still had remains of meals in them, cow and chicken bones, grain, lentils, and grape seeds. The town must have been predominantly Mussulman; it was deserted in a great hurry, probably due to a raid by an Afghan king about A.D. 1750. Some pottery is identical with that used at present, but not all modern types are there and there are some different shapes. The painted decoration is a brilliant design of a late period. Textiles and coins and other metal objects were also found. Hundreds of fragments of glass finger rings and bracelets were found, in an inexhaustible variety and brilliance of coloring. They had a base with a pattern of colors worked on by the intarsia technique. Many beads of glass, clay, and semiprecious stones were also found. W.D.F.

Fukien, China's rich new field for archeology. MALCOLM F. FARLEY. *Asia*, **39**, 641–46 (1939).—A kiln site near Lingnan University in Canton was used for centuries. The pottery ranges from the variety made during the pre-Han dynasty (coarse, handmade) to an advanced wheel-made variety. It forms a connecting link between nearby culture centers. At Chaochowfu, near Swatow, is the most extensive kiln site dump seen in China. It was active in the T'ang dynasty and until the end of the Sung and Yuan periods. Ware from this site was exported as far as Syria and Egypt. On the Min River, excavations showed Ying Ch'ing ware of endless variety, Yüeh ware, many other known and unknown types, and an older ware than had before been known. At Tehwa is the site of the manufacture of blanc de chine. It was sent to the far parts of the earth and exerted tremendous influence on ceramics in Europe. Near Chuanchowfu is a village where dragon-decorated jars are now made and have been made for over 1000 years. These jars have been shipped extensively to the South Sea islands and are found in excavations there. Excavation in the Foochow city dump, in use for 2000 years, yielded a ton or so of ceramic material. At four different sites a pre-Han culture has been found including handmade pottery and stone axes. Fukien potters have been predominant in Chinese ceramics. All over the Near East this Chinese pottery can be found. W.D.F.

German glassmakers in old Norway. BRUNO ROEMISCH. *Sprechsaal*, **73** [8] 63 (1940).—The Norwegian glass industry is now 200 years old. The first glassmakers were artisans summoned from Germany, chief among whom was Georg von Langen of Braunschweig who went to Norway in 1739 to organize the industry. The first glass factory was built at Aas, and soon afterward other plants were built. M.V.C.

Moses Golding. EDWIN GOLDING. *Bull. Amer. Ceram. Soc.*, **19** [6] 205–206 (1940).

Ku Yueh Hsüan vases in a porcelain exhibition. HELEN COMSTOCK. *Connoisseur*, **105** [463] 120 (1940).—At the Galleries of Tenying, in an exhibit covering a period from the Sung to the late 18th century, is a pair of *Famille Rose* vases of unusual quality. They are ascribed to Ku Yueh Hsüan, the director of the imperial factory, in the period of the Emperor Ch'ien-lung; they are $7^1/_2$ in. high and show the four-character Ch'ien-lung mark in red. The clear enamel colors and their skillful application represent the ultimate in Chinese porcelain decoration. Illustrated. M.E.P.

Reproductions and fakes of English 18th-century ceramics. WALLACE ELLIOT. *Trans. English Ceram. Circle*, **2** [7] 67–82 (1939).—A reproduction is a copy intended to deceive while a fake is a genuine piece altered to enhance its selling value, the reproducing or faking in each case being done after collectors have taken an interest in the articles. Reproductions and fakes are divided into eight kinds. Each is discussed and examples are given which are all illustrated. Reproductions are detected by the examination by experts of the suspected and genuine ware, attention being paid to modeling, glaze color, weight, artistic qualities, appearance of the mark, etc. In nearly all cases, faking necessitates refiring, which can be detected if the article has been in use some years. Absorbed moisture or grease in the paste or glaze permits the sulfur in the coal to combine with the lead present in most older pottery, making the glaze dull or even iridescent. Furthermore, smoke penetrates where the glaze is not continuous. If smoke is not present, the moisture on refiring causes the glaze to bubble up and become rough. The glaze may devitrify, taking on a mat or iridescent appearance. Other means of detecting fakes include searching for scratches running under the decoration, comparison of the decoration with that of genuine ware, and the use of ultraviolet light in the absence of visible light to detect restoration of missing handles, etc. 45 photographic illustrations. L.R.B.

Shapur, city of kings. R. GHIRSHMAN. *Asia*, **39**, 716–20 (1939).—Shapur I succeeded to the throne in A.D. 242 and built the city of Shapur as a royal residence, not a capital. The great stone palace was decorated with stucco. This decoration is distinctly Hellenistic in inspiration and must be due to artists brought from the West. W.D.F.

Soviet archeology today: I. HENRY FIELD AND EUGENE PROSTOV. *Asia*, **40**, 272–77 (1940).—A review is given of recent Soviet work at Phanagoreum, Kerch, Myrmekion, Olvia, and Eski-Kermen in the Black Sea area; at Tianeti, Dzherulsk, and Tsalka in Georgia; and Makhmudzhug and Dzhuga in Armenia. **II.** *Ibid.*, pp. 327–30.—The authors describe work at Kunya-Urgench and Anau (Turkoman); Samarkand, Bukhara, Varakhsh, the Sok river, and Uch-Kurgan (Uzbek); Lake Bailikul and Alma-Ata (Kazak); Talas Valley (Kirghiz); and Pazyryk (Siberia). W.D.F.

Stained glass. JAMES H. HOGAN. *Jour. Roy. Soc. Arts*, **88** [4560] 569–85 (1940).—H. points out that glass staining has no relation to picture painting but is an art in itself, dealing with the transmission of light through colored material; in painting, a colored pigment is applied to a flat surface upon which the light is reflected. A stained-glass window is an essential part of a wall and should conform to its surroundings. The grooved leads holding the pieces of glass together have a design value as well as a practical one. The designer of stained glass must know

and utilize color harmonies and good patterns, but his most difficult problem is to master the subject of light. Artistic and practical suggestions for coping with this problem are offered. The making of colored glass and its history and prominent eras are given with discriminating detail. Sources of design and subjects for representation are amplified; the extension of stained glass from religious subjects and churches to rich private homes and its temporary decadence in the 19th century are noted. The current period of revival is comparable to great periods of the past. Many windows of recent date compare favorably with those of medieval origin. Designers and craftsmen in Great Britain, the Continent, and the U. S. are doing work of great merit. The Pavilion of Stained Glass in the Paris Exhibition of 1937 is mentioned as being both exciting and stimulating in its revelation of ingenious devices and practical, as well as artistic, effects. A 13th-century window in the Cathedral at Bourges, another in Ockwell manor, Berks, and H.'s own window in the great central space of Liverpool Cathedral (*Ceram. Abs.*, **19** [2] 39 (1940)) illustrate his interpretative comments upon the historical perspectives of the art. K.R.

Test diggings in Syria. ROBERT J. BRAIDWOOD. *Asia*, **40**, 47–52 (1940).—Excavations were made at the Tabbat al-Hammam and Tell Simiriyan mounds to locate the ancient city of Simyra, a Phoenician city equal in importance to Tyre and Sidon. Tabbat al-Hammam is on the coast 28 miles north of Tripoli; it was occupied from 500 B.C. to Byzantine times. A small series of early Iron Age pottery was found, including some Byzantine red pottery, Cypriote trade pieces, and an interesting series of clay figurines. There was a long hiatus below this; the oldest culture represented is a Neolithic layer of 4000+ B.C., with potsherds corresponding to the earliest pottery found in Syria. These deposits show that the city of Simyra must be inland. The Tell Simiriyan mound three miles due east showed remains of a fired mud brick architecture and late Bronze Age pottery, as would be expected in Simyra. Another test pit down the slope showed middle Bronze Age potsherds like those found at Byblos and Ras Shamra. The lowest test pit showed potsherds of two phases of the early Bronze Age, a later calciform series and an earlier comb impressed series. The earlier also had reserved slip pottery of Judeideh XII and trickle painted pottery. Complete excavation of these mounds is recommended. W.D.F.

Village artists of prehistoric Iran. DONALD MCCOWN. *Asia*, **40**, 104–109 (1940).—The twin mounds of Tul-i-Bakun in southwestern Iran were excavated. The earliest people made crude, undecorated vessels. The new settlers introduced the painting of vessels, using various zigzag and geometric designs; birds were also used. The settlement was changed to the other twin mound. The people were peaceful, with few weapons. Groups of houses were made in contact with each other. The use of metal was known. Tools, as well as toys, games, and figurines, were made of pottery. Seals were made to identify property. The artistry of the people developed tremendously. It is hard to understand the interrelation of the early cultures in Iran because of few excavations and the regional differentiation of basically similar people. This culture was ended by the invasion of a people who made plain red-burnished pottery. This was about 4000 B.C. W.D.F.

Virtues of English pottery. BERNARD RACKHAM. *Trans. English Ceram. Circle*, **2** [7] 99–111 (1939).—Features of English pottery and porcelain up to the beginning of the 19th century are (1) fitness for purpose, (2) utilitarian ornamentation, e.g., the top of a jug handle is grooved with two fingers like the bottom of the handle in smearing it into the body of the jug, (3) adaptation of foreign methods to suit native methods and taste rather than slavish imitation, and (4) relative avoidance of painted decoration for which English potters have not shown as much aptitude as Continental potters. Wedgwood queen's ware set a new standard to which the products of Leeds pottery can be compared. This ware owes very little to foreign influence. Among English porcelain, Worcester and Derby are pre-eminent. In later ware, the influence of Meissen and Sèvres is noticed. 9 illustrations. L.R.B.

Youthful Bacchus. *Coronet*, **8** [2] 107–108 (1940).—A double-spread illustration in color depicts a ceramic statuette, from the Metropolitan Museum, created by Mr. and Mrs. Charles Vyse of Chelsea, England. See "Pottery. . . ," *Ceram. Abs.*, **18** [7] 171 (1939). M.E.P.

BOOKS AND SEPARATE PUBLICATIONS

Bible and Archaeology. FREDERICK KENYON. George H. Harrap & Co., Ltd., London. Price 15s. Reviewed in *Times Lit. Supp.*, **39** [1998] 240 (1939).—K. presents, for nonspecialist readers, the principal results of archeological research insofar as they relate to the Bible. He begins with the excavations of Layard at Nineveh and breaks off at the point where work has been suspended as the result of the present war. Illustrated. K.W.W.B.

Black Pottery of Coyotepec, Oaxaca, Mexico. PAUL VAN DE VELDE AND HENRIETTE ROMELIKE VAN DE VELDE. Southwest Museum Papers, No. 13. Southwest Museum, 42 pp. Price 75¢. Reviewed in *Science News Letter*, **37** [8] 128 (1940). P.G.H.

Early Pottery of the Jebeleh Region. ANN M. H. EHRICH. American Philosophical Society, Philadelphia. 129 pp. Price $2.00. Reviewed in *Science News Letter*, **37** [8] 128 (1940). P.G.H.

Transactions of the Oriental Ceramic Society, 1938–1939: Vol. XVI. Shenval Press. Price 21s. Reviewed in *Times Lit. Supp.*, **39** [1992] 175 (1940).—The following papers are included: "Chinese ceramics: a retrospect," by R. L. HOBSON; "Excursion to Ching-tê-Chên and Chi-an-Fu in Kiangsi," by A. D. BRANKSTON; and "Technical notes on Chinese bronzes, with special reference to patina and incrustations," by H. J. PLENDERLEITH. Illustrated. K.W.W.B.

PATENTS

Designs for:

Bottle. THOMAS D'ADDARIO (Tahiti Corp.). U. S. 120,663, May 21, 1940 (Feb. 15, 1940). J. L. DUNNOCK (Swindell Bros., Inc.). U. S. 120,804, May 28, 1940 (March 28, 1940). K. C. FAY. U. S. 120,668, May 21, 1940 (March 16, 1940). B. D. FUERST (Owens-Illinois Glass Co.). U. S. 120,806, May 28, 1940 (April 3, 1940). M. S. IREYS (Armstrong Cork Co.). U. S. 120,846 and 120,847, May 28, 1940 (March 13, 1940). M. W. LOWENSTEIN. U. S. 120,624, May 21, 1940 (Jan. 25, 1940). U. S. 120,625, May 21, 1940 (March 8, 1940). O. S. PARKER (Cameo Vineyards Co.). U. S. 120,677, May 21, 1940 (April 3, 1940).

Centerpiece bowl. R. R. KOSTELLOW (United States Glass Co.). U. S. 121,119, June 18, 1940 (Feb. 5, 1938).

Combined vase and candlestick. R. A. MAY (Duncan & Miller Glass Co.). U. S. 120,647, May 21, 1940 (March 1, 1940).

Glass bottle. L. J. KIHN (J. T. & A. Hamilton Co.). U. S. 120,813, May 28, 1940 (Feb. 20, 1940).

Glass jar. W. J. LUNDY (Olean Glass Co.). U. S. 120,815, May 28, 1940 (March 12, 1940).

Glass jug. A. J. DREITZER (Federal Glass Co.). U. S. 120,614, May 21, 1940 (March 29, 1940).

Vaporizer. F. E. OWEN. U. S. 120,871, June 4, 1940 (Dec. 18, 1939).

Cements

Calorimetric studies of the system SiO_2–CaO–H_2O. VITTORIO CIRILLI. *Ricerca Sci.*, **10** [5] 459–61 (1939).—The heat of solution, in calories per gram, of CaO was plotted against the ratio CaO/SiO_2, dry and hydrated. The products obtained with ratios of CaO/SiO_2 of 1 and 1.5, dried at constant weight in vacuum over P_2O_5 and

KOH, had the following compositions, respectively: $CaO \cdot SiO_2 \cdot H_2O$ and $3CaO \cdot 2SiO_2 \cdot 3H_2O$. See "Pozzuolanic. . .," *Ceram. Abs.*, **19** [4] 86 (1940). V.S.M.

Hydrated calcium aluminates. VITTORIO CIRILLI. *Ricerca Sci.*, **10** [6] 559–62 (1939).—The heat of solution and the hydration of $3CaO \cdot Al_2O_3$, $2CaO \cdot Al_2O_3$, and $4CaO \cdot Al_2O_3$ were studied. The behavior of $3CaO \cdot Al_2O_3 \cdot 6H_2O$ is similar to that of $2CaO \cdot Al_2O_3 \cdot 5H_2O$. For these aluminates the first 5 mol. of H_2O are present as water of constitution, and for the $4CaO \cdot Al_2O_3 \cdot nH_2O$ series 8 mol. of H_2O are water of constitution; the remainder is held as in zeolite. V.S.M.

Hydration of $3CaO \cdot Al_2O_3$. O. E. RADCZEWSKI, H. O. MÜLLER, AND W. EITEL. *Naturwissenschaften*, **27** [50] 837–38 (1939).—The hydration of $3CaO \cdot Al_2O_3$ at room temperature may result in two hydrates containing either 12 or 6 mol. of H_2O. Very fine air-dried powder was shaken for 16 hr. in water at room temperature. With the electron microscope (31,000×), hexagonal crystals of $3CaO \cdot Al_2O_3 \cdot 12H_2O$ were found with a maximum length of 1.3, a width of 0.9, and a thickness of 0.05 to 0.1 micron. In a mixture of isobutyl alcohol with water (18% H_2O), $3CaO \cdot Al_2O_3 \cdot 6H_2O$ was formed at room temperature; the crystals looked spherical, but they were supposedly icositetrahedrons 0.06 to 0.13 micron in length. Besides these crystals, others were found which had the same dimensions and shape as the crystals of $3CaO \cdot Al_2O_3 \cdot 12H_2O$ in the first test. J.M.N.

Hydration of $3CaO \cdot SiO_2$. O. E. RADCZEWSKI, H. O. MÜLLER, AND W. EITEL. *Naturwissenschaften*, **27** [48] 807 (1939).—Air-dried $3CaO \cdot SiO_2$ was shaken in water for 2 hr. at room temperature. Nodules and $Ca(OH)_2$ shaped like half spheres 0.2 to 1.0 micron large were then found with the electron microscope (31,000×). In an isobutyl alcohol-water mixture (18% H_2O) after 2 hr., the half spheres were again found (only 0.1 to 0.2 micron large), together with disks 0.07 to 0.1 micron in diameter. Besides these two, nodules of calcium hydrosilicates were visible. J.M.N.

System SiO_2–CaO–H_2O. VITTORIO CIRILLI. *Ricerca Sci.*, **10** [11] 1042–43 (1939).—By studying the progressive hydrolysis of tricalcium silicate at 23°C. with a calorimeter and the products obtained with X-rays, C. proves that the interpretation by Bessey (*Proc. Symposium Chem. Cements, Stockholm, 1938*) is incorrect. The products obtained with a ratio of CaO/SiO_2 of 1.5 are present as $Ca(OH)_2$ and $3CaO \cdot 2SiO_2 \cdot aq$. V.S.M.

PATENTS

Manufacture of Portland cement. I. C. BECHTOLD (California Portland Cement Co.). U. S. 2,203,809, June 11, 1940 (March 18, 1938).—The process includes grinding Portland cement clinker admixed with a small percentage of a substituted glycerol to produce Portland cement in finely divided form.

Producing white cement. OTTO SCHWACHHEIM. U. S. 2,201,143, May 21, 1940 (June 21, 1938).—A process of producing white Portland cement by adding substances giving off free chlorine to the raw material consists in mixing chloride of lime with the raw material, heating the mixture in a reducing atmosphere until sintering occurs, and cooling it while excluding air.

Enamel

Adherence phenomena of sheet iron ground coats. E. E. HOWE. *Better Enameling*, **9** [9] 13–19 (1938).—H. discusses in detail the tests made in an effort to explain the mechanics of adherence and gives their results. Practically all the experiments described indicate that the adherence of a sheet iron ground coat to enameling iron is due to an oxidation of the iron with subsequent solution in the enamel to form what might be termed a solution or a chemical union bond. Nickel functions in this operation as an inhibitor of the formation of too heavy an initial scale which must be taken up by the molten enamel. Cobalt oxide increases the solubility of the initial scale in the enamel and also keeps the relationship between solubility and precipitation in balance. Numerous photomicrographs are given. J.J.H.

Black and colored ground enamels. H. LANG. *Glashütte*, **70** [9] 119–20 (1940).—Enamels which are fired only once usually show cracking or fish-scaling; these are caused by a thicker coat and a lower firing temperature than are customary for ground enamels; such enamels do not have the appearance of enamels fired two or three times. The "spotted" enamels are the most successful of the enamels fired once; they have a good luster and appearance. There are two processes for making enamel coats which are fired once: (1) Ordinary ground enamel of the usual thickness is dried, heated, sprayed with a thin coat (powdered) of a good black, blue, or other colored enamel, and fired as an ordinary ground coat. (2) In this process, derived from the "tiger-spotted" enamels, a certain amount of cobalt sulfate solution is added to the enamel slip, and the enameled object is sprayed uniformly with a warm, rather concentrated soda solution. In this case the enamel must be preopacified with cryolite, antimony, or ceric oxide, and the color must not be too dark. Firing is the same as that for ground enamel; the sharper the firing, the better and larger is the spot formation. M.V.C.

Effects of variables on efficiency of ball-mill operation. R. L. FELLOWS. *Better Enameling*, **9** [2] 10–13 (1938).—F. presents a systematic study of the variables in the operation of ball mills. The tests conducted consisted primarily of varying (1) amount of frit charge, (2) amount of ball charge, (3) size of balls used, (4) water content, (5) pressure, (6) temperature, (7) speed of the mill, and (8) amount of enamel left in the mill. Conclusions are as follows: (1) the ball charge should always be kept at 55 to 60% of the volume of the mill; (2) the water content should be controlled carefully; (3) unground frit is probably caused by too small balls or too many undersized balls; (4) temperature has no effect on the efficiency of ball-mill operation, but too high a temperature has a detrimental effect on the set and working properties of the enamel; the temperature should be from 70° to 90°F.; (5) the correct frit charge for a certain mill can be determined and should be adhered to; undercharging of the mill is hard on the lining; (6) the speed should be kept fairly constant. Numerous charts are given. J.J.H.

Enamel. A. DIETZEL. *Sprechsaal*, **73** [8] 63–64 (1940).—D. discusses the term "enamel" as defined by other technicians and proposes that only those substances which can be defined as follows should be called enamels: an enamel is a hardened glassy substance obtained by fusing or fritting which has an oxidic ground composition and which is melted in one or more layers on a piece of metal. M.V.C.

Factors affecting rate of nickel deposition in nickel pickling. J. M. ZANDER. *Better Enameling*, **9** [6] 5–8 (1938).—Z. discusses the factors controlling the amount of nickel deposited from a nickel bath under various conditions. Results of numerous tests are given, together with charts showing the effect of variables on the amount of deposition. J.J.H.

Fine grinding and thin cover coat application. ANON. *Better Enameling*, **9** [12] 5–7 (1938).—Literature regarding the fine grinding of cover-coat enamels is reviewed. Charts and references are given. J.J.H.

How enameling sheets are made today. A. C. BADGER. *Better Enameling*, **9** [6] 12–15 (1938).—B. discusses the process of making enameling sheets, from the initial selection of raw materials of exactly the right analysis to the final treatment and testing of the finished sheet surface. Numerous tests are given. Illustrated. J.J.H.

Influence of drying methods on tearing. E. E. HOWE. *Better Enameling*, **10** [12] 13–15 (1939).—H. discusses three types of drying: (1) circulating hot air, (2) radiant heat without forced draft, and (3) still air. Various experiments are described, and it is pointed out that finer

grinding increases tearing. The specific gravity of the slip and the method of use of the spray gun are factors in tearing. Electrolytes, except sodium nitrate, increase tearing. H. suggests that drying should be arranged so that evaporation takes place from the metal outward and surface drying is avoided. J.J.H.

Matching synthetic whites and porcelain enamels. F. H. EMERY. *Enamelist*, **17** [6] 7–12 (1940).—There are two types of color matches: (1) the absolute (dead) match which is good under any illumination and which is tested with the Hardy spectrophotometer, and (2) a drifting match (nonspectrophotometric) which is good only under a specific illumination. Variations in spray weight and firing may cause changes in the reflectance of enamels. A few general rules and examples for producing a dead match are given. 4 diagrams. L.E.T.

Mechanical setup required to use finer ground cover coats successfully. F. DOERING. *Better Enameling*, **10** [12] 6–8, 22–23 (1939).—For successful use of thinner cover coats of the latest type of superopaque enamels, it is necessary to eliminate defects in the sheet steel and ground coat. Methods and equipment are described. J.J.H.

Nickel dip in enameling plants. ANON. *Ceram. Ind.*, **34** [6] 52–54 (1940).—The theory and practice of the use of the nickel dip in enameling plants are reviewed, and a chart is presented to show actual operating conditions, including type of nickel salts, strength of solution, temperature of dip, time, pH range, and cost, as determined in a number of commercial plants. The nickel dip improves ground-coat adherence, eliminates much copper-heading, greatly decreases fish-scaling, and gives a more uniform surface texture and a better fired color. It has the following disadvantages: the tank requires careful control, there is an added cost of about 15¢ per 1000 sq. ft., and the repickling of nickel-dipped ware is difficult. Illustrated. L.M.C.

Opacity changes resulting from enamel of different specific gravities. E. E. HOWE AND L. A. LANGE. *Better Enameling*, **9** [4] 19–22 (1938).—Tests conducted with high and low specific gravity enamel slips are discussed. An increase in the specific gravity of the milled enamel gave a corresponding increase in the reflectance of the fired panels. The increase in opacity was approximately the same for the three types of frits tested, indicating that the composition of the enamel had but little effect on this property. The increase in opacity resulting from application of the milled enamel at a high specific gravity is due to voids which are converted into bubbles upon fusion of the enamel. Changes in specific gravity had very little effect on abrasion resistance and surface gloss. Numerous graphs are shown. J.J.H.

Pickling tanks for the enameler. H. BAILEY. *Enamelist*, **17** [7] 10–12 (1940).—B. discusses the different types of pickling tanks and materials used in their construction, such as acidproof stoneware, brick, concrete, carbon blocks, Monel, alloy steels, lead, plastics, rubber, sulfur cements, wood, and stone. Illustrated. L.E.T.

Porcelain enamel manufacture. CHARLES J. MORSE. *Ind. Gas*, **18** [10] 5, 6, 14 (1940).—Continuous and batch gas-fired smelters are used by the Chicago Vitreous Enamel Products Co. in producing enamel frit. Raw materials are automatically weighed, and the smelting temperature is automatically controlled. The frit then goes to a gas-heated rotary drier. S.S.

Porcelain enamel strengthens metal. D. R. GOETCHIUS. *Enamelist*, **17** [8] 13–14 (1940).—Tensile strength specimens were made of standard enameling stock of 8, 10, 12, and 14 gauges; these were then pickled and enameled with one ground coat and one cover coat, giving an average enamel thickness of 0.011 in. The average yield points of the unenameled specimens pulled to failure are as follows: 14 gauge, 32,970; 12 gauge, 32,895; 10 gauge, 30,605; and 8 gauge, 45,270 lb. per in.^{2}. Average yield points for enameled specimens pulled until failure are as follows: 14 gauge, 43,600; 12 gauge, 32,500; 10 gauge, 29,100; and 8 gauge, 40,500 lb. per in.^{2}. L.E.T.

Rapid determination of quartz in feldspar. G. H. MCINTYRE AND M. BOZSIN. *Ind. Eng. Chem., Anal. Ed.*, **12** [6] 326–28 (1940).—In the chemical analysis of feldspar, all of the silica is determined as such, but no determination is made of the amount of quartz. As quartz and feldspar are major ingredients in vitreous enamels and the ratio between the combined silica and quartz in feldspar is variable, the quantity of quartz present in feldspar must be determined in order to ensure uniformity of enamel composition. A method is described for the determination of quartz in feldspars which is rapid and requires no special apparatus for sample preparation and grain-diameter measurements. It has been standard in the analytical laboratories of the Ferro Enamel Corp. for over 10 years and has been proved to be practical. It is based upon the principle that unfused quartz is anisotropic and that, when a powdered vitreous mixture is viewed under a polarizing microscope with crossed Nicols, the number of quartz grains in a definite area may be counted. These will appear light or colored, whereas the glass will be dark. Quartz grains which are in the extinction position will remain dark when so examined, but as the standards are examined in the same manner the error is nullified. Illustrated. F.G.H.

Relation of defects in enamel coatings to hydrogen in steel. C. A. ZAPFFE AND C. E. SIMS. *Jour. Amer. Ceram. Soc.*, **23** [7] 187–219 (1940).

Seeger again modernizes. D. CHASE AND E. GOETZ. *Better Enameling*, **11** [1] 3–7 (1940).—A modern continuous plant which enamels refrigerators is described. Reference is made to the equipment and precautions necessary for finishing the ware in one cover coat. Illustrated. J.J.H.

Testing the properties of cooking utensils for electric cooking stoves. HUGO SCHULZE. *Glashütte*, **70** [8] 93–96 (1940).—To obtain the greatest efficiency from electric cooking stoves, cooking utensils must be heavier than those used with other types of stoves. In the manufacture of utensils for electric stoves, important factors must be taken into consideration: the construction and form have as much effect as casting, smelting, and the composition of the iron, and the preparation and suitability of the enamel are equally important. The requirements for enameled cooking utensils for use on electric stoves were determined by (1) the determination of the measurements and the description of the utensils, (2) tests on surface variations, (3) determination of the degree of efficiency and boiling time, (4) tests on the change of form, (5) repetition of 3, and (6) testing handles. M.V.C.

Training for the porcelain enameling industry. J. T. IRWIN. *Enamelist*, **17** [6] 25–26 (1940).—I. calls attention to the local trades schools as a medium for training porcelain enamel personnel. L.E.T.

Use of vitreous enamel tubes in warm water pipe system. G. C. KORNMESSER. *Gesundh.-Ing.*, **61** [17] 224 (1938).—Tubes of hard porcelain 1.5 m. long, 65 mm. in diameter, and with an insulation of 1 m. are used. The system works very satisfactorily. See *Ceram. Abs.*, **17** [7] 256 (1938). W.D.K.

What we have learned about enameling clay since World War I. J. T. IRWIN. *Enamelist*, **17** [8] 5–12 (1940).—While the imported Vallendar clay has been used as the most satisfactory enameling clay for many years, variations in setting-up behavior and impurities (sand, marcasite, wood fiber, etc.) were found quite often. Steps were taken to purify, dry, and blend this clay; a better understanding of the general requirements and type of enameling clay was obtained at the same time. This will make it possible to duplicate Vallendar clay with domestic clays and, in time, eliminate all imported enameling clays. L.E.T.

White arsenic in the enameling industry. M. DECHIGI. *Arch. Gewerbepath. & Gewerbehyg.*, **7**, 468–76 (1936).—The majolica industry is one of the oldest in Italy, recalling many cities famous for its production and the name of an illustrious painter, Della Robbia. To preserve the beautiful white of this well-known ware, Della Robbia advised the use of "calcina," a mixture of tin and lead oxide. The

union of the two metals produced oxidation in which the lead gave up its oxygen. The tin oxide then functioned as the white pigment in clearing up the clouded majolica. The development of industrial hygiene eliminated the lead procedure and brought about direct use of tin oxide in its pure condition. D. points out that the use of tin oxide greatly improved the enamel scientifically, practically, and hygienically. In spite of these advantages, substitutes were sought to cheapen the cost of production. Antimony oxide and white arsenic (As_2O_3), anhydrous arsenical acid) were tried out, although both were recognized at once as being poisonous substances and very undesirable for the composition of many articles of household use. D. mentions three fatal cases reported in England which were due to drinking lemonade from iron containers enameled with antimony oxide (*Ceram. Abs.*, **12** [6] 216 (1933)). Increasing use of antimony oxide in Italy caused recognition of the fact that its toxic effects were equal to those from glazing utensils with lead oxide, but no striking cases occurred there. This recognition was embodied in an industrial hygiene ordinance in April, 1927. The attention of enameling industries, however, was called not so much to antimony dangers as to the need of regulation and control. The use of anhydrous arsenic acid as a substitute for the harmless tin oxide in enameling tile, household articles, and sanitary conveniences for bathrooms is discussed. Because of its cheapness the arsenic medium was increasingly used for majolica tile and glass. The danger in arsenic materials is stressed, and current inconsistencies in their use are shown; though Paris green is no longer used in textiles, the walls of dwellings are frequently painted or papered with colors having an arsenic content. Paris green is also in use in malaria-infested regions for control of mosquito larvae and is often found about schools and homes. Arsenic is present in various sprays used against parasites and flies and in mixed dusts. Physicians concerned with industrial hygiene in the U. S. first noted the increasing use of arsenic trioxide in the enameling industry (International Labor Bureau, 1935). D. discusses the industrial techniques in which such use of arsenicals may result in household dangers, not alone its use for tile but also for the enameling of water kettles and kitchen utensils where the arsenic may be brought directly into contact with food. He lists the symptoms of arsenical poisoning arising from domestic usage: headache, throat inflammation, coughs, dyspepsia, diarrhea, and skin inflammations. The difficulties in distinguishing occupational poisoning from that derived in homes are pointed out. In his tests for arsenic as found in tile and utensils, the biologic method was used; the procedure is given: the breakdown of the arsenic compound through hypomycetes, *Pencillium brevicaule*, in combinations according to Gosio, and resulting in the characteristic odor of garlic, also of fungi. The chipping-off of tile in constant use and from continual washing yielded arsenical results in these tests. D. concludes that the use of arseniated enamels is highly undesirable and should be under the same inspectional control as antimony oxide or a lead content enamel. 16 references. K.R.

White ground enamels for iron. K. P. Azarov and N. S. Kharchenkova. *Zhur. Priklad. Khim.*, **12** [11] 1598–1600 (1939).—Adherence is considerably improved by covering the iron with a nickel layer and by using a zirconium opacifier mixed with antimony oxide. A white ground coat can, in many cases, be substituted for the usual cobalt ground coat; this simplifies the technological process. P.B. & E.S.

SEPARATE PUBLICATION

Porcelain Enamel Colors and Color Matching. Porcelain Enamel & Mfg. Co., Baltimore, 1940. 13 pp. Price $1.00.—The method of matching a large number of colors with a small color inventory is described. Tables showing factors which affect color are included. Simple devices to be employed in achieving good color-matching results are suggested. The book contains three pages of color samples, including 16 basic and 22 blended oxides and 10 Bureau of Standards colors, known as Kitchen and Bathroom Colors, determined through the collaboration of leading department stores, manufacturers, and the U. S. Bureau of Standards. The book also tells how 400 colors may be produced, using only 16 basic oxides, and provides useful formulas for the purpose.

PATENTS

Enameling method. J. E. Rosenberg. Ger. 686,290, Dec. 14, 1939 (April 24, 1936); VI/48*c*. 1.—In an enameling process, the surfaces to be enameled are given an interrupted or discontinuous coating, and the surface not covered by this preliminary coating is etched. The protective coating is produced as a metallic netlike coating. A different kind of etching compound is employed. D.A.B.

Equalizer support for enameled tanks during firing. E. H. Nilson (A. O. Smith Corp.). U. S. 2,201,786, May 21, 1940 (June 18, 1937).

Molded product. A. B. Ruddle. U. S. 2,204,913, June 18, 1940 (Sept. 25, 1937).—A fired molded product of sufficiently high tensile strength to be used as a core for metal casting purposes and having a high degree of resistance to heat and moisture comprises a mixture of sand and a binder which comprises an aqueous solution of an alkali-metal silicate, an alkali metal fluosilicate, and a compound of boron.

Glass

Binding glass batch. R. L. Shute. *Ceram. Ind.*, **34** [6] 38–39 (1940).—A survey was made of various binding agents and binding methods for glass batch, other than those used by the Ford Motor Co. and Sneath Glass, to see what economies could be effected. Cornstarch had fair adhesive power when dried at temperatures around 300°F. but was not equal to sodium silicate. Portland cement produced better results but required several hours to reach maximum hardness. Random lumps of batch mixed with sodium silicate and allowed to dry were firm enough to stand rough handling, without a high-pressure briquetting press. A recapitulation of costs showed that the Ford method of briquetting increased costs $60.40 a day for 80 tons of batch. Mixing and drying the batch in random lumps increased costs by $74.25 daily. Savings made possible in comparison with loose batch, however, amounted to a $9.00 saving in raw material carried into the checkers, a $10.00 saving in depreciation of checker brick, and $20.00 in depreciation on the rest of the furnace; the total daily saving was $39.00. The net increase over direct saving effected was as follows: briquetting, $21.40; mixing and drying (crushed limestone batch), $35.35; mixing and drying (powdered limestone batch), $27.25; and mixing and drying (burned lime batch), $24.05. Illustrated. L.M.C.

Calculation of the refractivity of glasses. Oscar Knapp. *Jour. Soc. Glass Tech.*, **24** [101] 37–40T (1940).—K. examines the accuracy with which the refractive index of glasses can be calculated from Gilard and Dubrul's constants (*Ceram. Abs.*, **17** [9] 301 (1938)), using data for 67 optical glasses. G.R.S.

Casti-ron molds for glassware. H. L. Walker. *Ceram. Ind.*, **34** [6] 45 (1940).—The normal and most common type of check in molds is perpendicular to the main axis and is due to a change of section; a second type involves no change of section and usually runs parallel to the main axis; and a third type is a network of checks which appears to be due to grain boundaries but is not. Tests were made on the checking characteristics of high- and low-strength sand-cast irons. The low-strength iron has a tensile strength of 20,000 lb. per sq. in. and a silicon content of 2.25%. The high-strength iron includes 1.75% silicon, 1.5% nickel, and 0.6% chrome and has a tensile strength of 54,000 lb. per sq. in. There was no free ferrite

present in the high-strength iron. An accelerated test involved heating to 1100°F. and sudden quenching in water at room temperatures. After as many as 40 cycles of this test, the iron was studied for checks, growth, and machinability. The quenching caused no change in microstructure. The low-strength iron showed more checks than the high-strength, but these were of less depth and smaller in magnitude. The high-strength iron had few checks, but they were much more severe in character. High-strength iron showed only about one-third as much growth during test as the low-strength. The low-strength iron after 40 cycles had a high pearlite content and showed better machinability. Another series of tests to compare chill-cast iron with sand-cast iron showed that (1) chill-cast iron after 20 cycles has about the same checking as sand-cast iron after 40 cycles, (2) low pearlite iron has checks of less severity than high pearlite but more of them, and (3) alloyed irons show less tendency toward heat checking than the purer irons, but they are usually harder to machine. L.M.C.

Chemical durability of glassware. A. HERMAN. *Ceram. Ind.*, **34** [6] 39–41 (1940).—H. outlines the reasons for specifying the chemical durability of bottles in which Joseph Seagram & Sons, Inc., pack liquors. After original experimental work was done, the powder test was discarded because it was too time-consuming, because of the difficulty of preparing samples, and because it was not completely reliable. It deals with the entire glass wall instead of the inside surface which is of particular interest. The present test is much shorter and involves the autoclave treatment of distilled water in a test bottle and then titration to determine the amount of alkalis leached out of the surface. See "Solubility. . .," *Ceram. Abs.*, **18** [8] 207 (1939). L.M.C.

Cutting glass tubes. ANON. *Mech. World & Eng. Record*, **107** [2785] 441 (1940).—Four simple methods of cutting glass tubes when a lathe is not obtainable are briefly described. F.E.S.

Effect of sustained loading on the breaking strength of sheet glass. A. J. HOLLAND AND W. E. S. TURNER. *Jour. Soc. Glass Tech.*, **24** [101] 46–57T (1940).—The effect of sustained loading on the modulus of rupture of specimens of sheet glass having round and well-polished edges and the dimensions 10 x 0.8 x 0.275 cm. was determined by loading the specimens to the extent of 100, 90, 80, 70, 60, 50, 40, and 30% of the previously determined mean breaking load and measuring the time interval between loading and fracture. For loads equal to or greater than 70%, a number of specimens fractured before the full load was reached. For loads less than 70%, some of the specimens remained unbroken after 1000 hr., the number unbroken increasing as the load decreased until all the specimens remained unbroken after 1000 hr. at the 30% load. For those specimens fracturing, a straight-line relationship held between the logarithm of the mean breaking time and the logarithm of the applied stress. A factor of safety of not less than 4 was suggested for sheet glass of the type described. G.R.S.

Glasses, organic and inorganic. HARRY MOORE. *Jour. Soc. Glass Tech.*, **23** [100] 347–71T (1939).—A definition of glass and a scheme for the classification of glasses are discussed. The structure of inorganic and organic glasses is outlined, and their physical properties are tabulated. G.R.S.

Glass filters of known uniform pore size up to the dimensions of colloidal particles. JOHANN BERNDEL. *Kolloid-Z.*, **90** [2] 194–96 (1940).—Filters which must be resistant to alkalis, acids, pressure, and heat are made of glass, porcelain, or other clay bodies. In colloid chemistry, filters with extremely small pore space are used. B. describes a method for producing extremely fine filters of glass. A number of fine tubings are welded together and reduced in size by heating and repeated drawings. An example is given for a glass rod having 117,649 uniform size pores (straight-lined). This rod is 1 mm. in diameter. Such rods can be cut into disks to make filters, some of which have the dimensions of colloidal particles. 1 photomicrograph and 1 table are given. L.E.T.

Glass for mechanical parts. T. J. THOMPSON. *Product Eng.*, **11** [5] 196–98 (1940).—T. explains the excellent properties of borosilicate glass and gives examples for replacing metal parts by it. Its thermal expansion coefficient (0.0000018 per °F. between 19° and 350°F.) is only $^1/_3$ to $^1/_4$ that of most metals. It has the following properties: modulus of elasticity 9.8 × 10^6 lb. per sq. in., specific gravity 2.23, specific heat 0.20, refractive index 1.474, thermal conductivity 8.1 B.t.u. per sq. ft. per hr. per 1 in. thickness per °F. at 77°F., dielectric strength in air 3200 kv. per in. at $^1/_{16}$ in. thickness, and electric volume resistivity 4 × 10^{14} ohm per cu. in. at room temperature. The abrasion resistance is about twice that of plate glass. Borosilicate glassware is transparent to certain wave bands of thermal radiation; this sometimes compensates for the low thermal conductivity and makes relatively high rates of heat transfer possible from high temperature radiation by a flame or by electric heating elements. The noncrystalline structure of glass very likely accounts for the notable absence of fatigue under repeated loading; a glass spring $1^1/_2$ in. in diameter has been made which exerts a force of 1 lb. at $^3/_{32}$ in. deflection. The dimensional variations of commercial borosilicate glassware are fairly small except in very small objects. M.H.

Increasing production in sheet-glass manufacture. RICHARD GENENGER. *Glashütte*, **70** [10] 133–35 (1940).—G. describes the means by which the production of plate glass according to (1) the "fixed table" method, (2) the Bicheroux process, (3) the Shuman process, (4) the Libbey-Owens sheet-glass machine, (5) the Fourcault machine, and (6) the Ford continuous machine can be increased. Increased production depends not only on the system of manufacture but also on the subsequent cooling system which in turn is dependent on the thickness of the glass and the allowable rate of cooling per second. After the glass has traversed the annealing zone, the rate of cooling can be increased. A table is given which permits the comparison of the capacity, size, output, production time, temperature decrease per second, cooling time, etc., of the six systems mentioned. M.V.C.

Influence of sodium chloride on the iron oxide content of molten soda-lime-silica glasses. R. HALLE AND W. E. S. TURNER. *Jour. Soc. Glass Tech.*, **24** [101] 41–45T (1940).—Glasses of the basic percentage composition SiO_2 73.5, Na_2O 16.5, and CaO 10.0, containing 0.08 per cent Fe_2O_3, were melted in platinum at 1500° in a gas-fired furnace, both without and with the addition of sodium chloride and certain other volatile materials. The amount of sodium chloride added in all cases was equivalent to 0.2% Cl in the glass. The other volatile materials added with the sodium chloride and their amounts in different series were (*a*) selenium, 0.4 oz. per 1000 lb. of sand, with cobalt oxide in appropriate amount to decolorize the iron oxide; (*b*) borax, to yield 2.0% in the glass; and (*c*) ammonium sulfate, 7.5 parts per 1000 of sand. Analysis of the resulting glasses showed that the losses of iron oxide in the presence of sodium chloride and other volatile constituents were (*a*) sodium chloride alone, 11.3%, (*b*) with selenium, 15.0%, (*c*) with boric oxide, 17.5%, and (*d*) with ammonium sulfate, 28.8%. G.R.S.

Installations for drying sand. K. H. BORCHARD. *Glashütte*, **70** [8] 96–98; [9] 115–19 (1940).—B. discusses the problem of drying sand for use in glass manufacture so that the quality is not affected and the construction of the drying device for the most efficient and economical operation. Differently constructed types of drying drums are discussed with regard to the relative cost of operation, efficiency, and improvements which could be effected. M.V.C.

Mass method of determining the general inclination of glasses to crystallization. I. I. KITAĬGORODSKIĬ AND T. N. KESHISHYAN. *Zhur. Priklad. Khim.*, **12**, 1309–10 (1939).—The crystallization process depends on the quantity of crystallization centers (*Kv*) forming in a unit of volume in a unit of time and on the rate of linear growth of crystals (*Ky*). The crystallization properties of glass must be judged not only by the latter but also by the effect characterized by the superposition sector of the *Kv* and

Ky curves. The tendency of glass to crystallize can be characterized by the relation of the crystalline and vitreous phases in the crystallization product of a considerable quantity of small samples held out at different temperatures during equal time intervals. P.B. & E.S.

Measuring cordiness of glass. FRANK W. PRESTON. *Ceram. Ind.*, **34** [6] 60, 62 (1940).—A new technique developed at Preston Laboratories presents the first quantitative measure of the cordiness of glass. The cords are removed from the parent glass and crushed to pass first 200-mesh and then 325-mesh screens. Dust is removed from the surface of the particles by washing in alcohol, and the moisture is removed by a high-temperature treatment in a special vacuum furnace. The cleaned fine particles are immersed in an organic solution which has a greater density than the glass itself. Sample tubes of this mixture are rapidly whirled in a specially built centrifuge operated under very closely controlled temperatures which can be measured and held within 0.02°. The solution is slowly heated until all particles, which formerly floated on the surface of the liquid, sink to the bottom. The temperature is very accurately recorded at the time when the first particles begin to sink and again when the particles have all settled to the bottom of the sample tube. The temperature interval during which this settling takes place gives an excellent measure of the cordiness of the original glass. The best commercial samples show a temperature interval of only $^1/_2$° from first to complete settling. The average commercial bottle contains some cords, and these give a test rating of about 2°. A range of 5° to 10° is recorded on ware which is poor in quality, and a remarkably high figure of 17° was determined for one sample of very inferior quality. L.M.C.

Mineral wool. A. GUTTMANN. *Tonind.-Ztg.*, **62** [63] 681–83; [64] 695–96 (1938).—G. gives a history of the development and a description of the manufacture of slag wool, rock wool, and glass wool. W.K.

Mortar and pestle for powdering glass. H. L. WUNDERLY. *Ind. Eng. Chem., Anal. Ed.*, **12** [5] 284 (1940).—The mortar consists of 5 in. of 1-in. iron pipe (nipple) on one end of which is screwed a pipe cap, loosely fitted in order to facilitate removal of the powdered glass. The pestle is a 16-in. length of a 0.75-in. iron rod, threaded at one end and fitted with a large iron nut having a diameter a little greater than 1 in. The nut is tightly adjusted to the rod and trimmed by a silicon carbide wheel to a size that will permit it to be inserted with ease into the mortar. The nut and rod are ground at the end until a flat pounding surface is attained. F.G.H.

New uses of glass. O. ULRICH. *Deut. Tageszig.*, May, 1939; abstracted in *Verre & Silicates Ind.*, **11** [3] 33–34 (1940).—Glass as a substitute for metal is now used for cooking utensils, fastenings for windows, door knobs, coat holders, curtain rods, water tanks, elements of refrigerators, printing cylinders, and in lamps with metal parts reduced to a minimum; it is also used to replace tin in canning, for glass faucets and glass piping for heaters and coolers, and in breweries, cold storage plants, dairies, chemical plants, and establishments for water cure. Pieces of glass piping up to 200 m. have been used, and the new profession of glass smith has appeared. Glass piping does not give any taste to liquors and does not favor the accumulation of carbonic acid. Glass is also used as a substitute for hard rubber storage batteries and receivers and, in construction, as glass brick and glass-insulating fiber. M.V.C.

North American glass industry in 1938. W. E. S. TURNER. *Jour. Soc. Glass Tech.*, **24** [101] 5–36T (1940).—While the productivity of the glass industry of the U. S. has continued to increase, the number of plants has decreased. A table gives the number of plants by years from 1899 to 1937. Production data for polished plate glass, window glass, and glass containers are given in three tables. Glass composition and raw materials are discussed, particular attention being given to the newer types of glass. Batch mixing and charging, refractory materials, fuels and furnaces, electric melting of glass, and glass-forming machinery are treated. Special features in the production of various kinds of glass include discussions of glass fiber products, glass building blocks, domestic glass, bottles, and containers. Scientific control and research in the industry are considered. G.R.S.

Progress in glass technology under the auspices of the Glass Delegacy during 1938–1939. ANON. *Jour. Soc. Glass Tech.*, **23** [100] 329–37T (1939). G.R.S.

Properties of laboratory glass of the Druzhnaya Gorka plant. K. V. MANZHURNET. *Zavodskaya Lab.*, **8**, 1009–10 (1939). P.B. & E.S.

Quartz glass as a material for the construction of chemical apparatus. F. A. KURLYANKIN. *Zhur. Khim. Prom.*, **16** [6] 37–39 (1939). P.B. & E.S.

Quartz glass with sharp cut-off at 2800 a.u. A. J. MADDOCK. *Jour. Soc. Glass Tech.*, **23** [100] 373–77T (1939).—By the addition of a small quantity of stannic oxide to quartz, a glass similar in its physical properties to pure fused quartz is obtained which sharply cuts off all radiation below about 2800 a.u. and which has a very steep transmission curve in this region. Its characteristics, methods of fusion, properties, and uses are discussed. G.R.S.

Substitution of NaCl for soda in glassmelting. N. P. KRASNIKOV. *Prom. Stroitel. Materialy*, **1939**, No. 1, pp. 129–34.—After reviewing the literature, K. suggests a method that has been preliminarily verified in semiplant conditions. The briquetted charge is melted at 1300° in a shaft kiln of the blast-furnace type. The melt is introduced from there into a refining basin where the refining takes place at 1450° to 1480°. The method is being investigated. P.B. & E.S.

Tank pressure and combustion control. I. G. FOWLER. *Ceram. Ind.*, **34** [6] 42–44 (1940).—Common subatmospheric pressure in glassmelting tanks results in a considerable infiltration of cold air into the furnace and regenerators. The first effect of such infiltration is a reduction in furnace temperature; the secondary effect is an increase in the quantity of waste gases and a decrease of their temperature. Both conditions impair the performance of the regenerators and increase the amount of fuel required to produce a given result. Infiltration of air also shortens the flame and creates a localized high temperature which produces a pool of hot glass adjacent to a pool of cooler glass. On the other hand, a few tanks are operated under high pressures; this results in exceptionally high repair bills and high fuel costs. An answer to these varying conditions lies in the use of furnace pressure control to allow a set ratio between air and fuel at all times. On a large tank, an orifice was placed in the combustion air line and in the gas line. A control damper was also placed in the air line to vary the amount of air depending upon the gas admitted to the burners. It is now possible to set the gas feed for any desired amount and rely upon the mechanism to give just the right amount of air at all times. This control serves to even the fire from one side of the tank to the other, regardless of the checker resistance. Illustrated. L.M.C.

Use of Na-Mg glasses for glass electrodes. A. PASYNSKII. *Zavodskaya Lab.*, **8**, 869–71 (1939).—For work with a glass electrode in the usual potentiometric scheme with a pointer galvanometer, glasses with the composition 28 Na_2O, 8 MgO, and 64% SiO_2 can be recommended. The measurements are accurate up to 0.03 to 0.5 pH in the interval pH 2.0 to 9.0. P.B. & E.S.

X-ray diffraction study of the structure of glass. B. E. WARREN. *Chem. Revs.*, **26**, 237–55 (1940); see "Nature . . .," *Ceram. Abs.*, **18** [5] 124 (1939). W.D.F.

BOOK

Glass (Glas): **Vol. II.** HERMANN THIENE. Gustav Fischer, Jena, 1939. 365 pp., 320 illustrations. Price 50 Rm.—T. discusses the chemical properties and the classification of glasses, viz., flat glass, hollow glass, and optical glass. A bibliography, a name index, and a subject index are included. For **Vol. I** see *Ceram. Abs.*, **10** [10] 691 (1931). M.V.C.

PATENTS

Acidproof container. J. H. SHERTS (Pittsburgh Plate Glass Co.). U. S. 2,202,675, May 28, 1940 (Feb. 25, 1939).

Annealing lehr and method of annealing glassware. W. S. RENDALL (Crown Cork & Seal Co., Inc.). U. S. 2,203,182, June 4, 1940 (Nov. 21, 1936).

Apparatus for drawing glass strip. PILKINGTON BROS., LTD., AND G. H. BAILLIE. Brit. 520,699, May 15, 1940 (Oct. 27, 1938).

Apparatus for pressing curved safety glass. WILLIAM OWEN (Pittsburgh Plate Glass Co.). U. S. 2,202,670, May 28, 1940 (July 13, 1938).

Apparatus for shearing molten glass. F. L. O. WADSWORTH (Ball Bros. Co.). U. S. 2,202,409, May 28, 1940 (Jan. 18, 1935; June 1, 1937).

Blowing form for producing hollow glass articles with base. WILHELM WAGENFELD, HANS HILLMANN, AND HANS BERGER (Vereinigte Lausitzer Glaswerke A.-G.). Ger. 685,220, Nov. 23, 1939 (Feb. 28, 1938); VI/32*a*. 8.—A blowing form consists of an upper form corresponding to the shape of the glass object and a rotary base form corresponding to the shape of the base. D.A.B.

Coated glass fabric. C. S. HYATT AND J. C. LOWMAN (Columbus Coated Fabrics Corp.). U. S. 2,204,859, June 18, 1940 (April 8, 1938; Sept. 14, 1938).—The process of making a decorative article of manufacture comprises producing a fabric of woven glass fibers, etching the fibers of the fabric, and applying to the fabric a liquid coating composition comprising the heat-reaction product of oil soluble phenol formaldehyde resin 100 parts, China-wood oil 312 parts, bodied linseed oil 160 parts, mineral spirits 200 parts, and titanium dioxide 300 parts in heated condition to the glass fabric structure so as to coat the fibers and penetrate the interstices between fibers, drying the coating to form a plastic flexible film interlocking the fibers together, and thereafter subjecting the coated fabric to a temperature of about 100° to 300°F. for a time sufficient to volatilize the thinners therefrom and produce a highly flexible decorative coated fabric composition.

Coating a glass tube with luminescent material. BRITISH THOMSON-HOUSTON CO., LTD., AND L. J. DAVIES. Brit. 520,937, May 22, 1940 (Oct. 3, 1938).

Container for molten glass. P. G. WILLETTS (Hartford-Empire Co.). U. S. 2,203,288, June 4, 1940 (April 29, 1936).

Edge-grinding apparatus. H. J. HAMANN AND C. B. WILLIAMS, JR. (Libbey-Owens-Ford Glass Co.). U. S. 2,205,009, June 18, 1940 (Aug. 18, 1938).

Frosted glass article and method of making. J. A. YUNCK (Corning Glass Works). U. S. 2,202,327, May 28, 1940 (June 7, 1938).

Gastight articles from quartz charge. ERICH LEONHARDT AND LUDWIG TEICHMANN (I. G. Farbenind. A.-G.). Ger. 680,370, Aug. 3, 1939 (April 14, 1937); VI/32*a*. 35.—Gastight articles are produced by adding gas-forming materials in small amounts to the quartz sand before casting the article. Gas is evolved during casting. D.A.B.

Glass-article bottomer. PAUL KRAUSE (Hudson Armstrong). U. S. 2,203,573, June 4, 1940 (Feb. 18, 1937).

Glass-cooling apparatus. JESSE GRANT (Corning Glass Works). U. S. 2,202,100, May 28, 1940 (Jan. 31, 1939).

Glass-to-metal seals. MARCONI'S WIRELESS TELEGRAPH CO., LTD. Brit. 520,942, May 22, 1940 (Oct. 30, 1937).

Heating of glass and other molten conductors in electrode furnaces. SOC. ANON. DES MANUFACTURES DES GLACES & PRODUITS CHIMIQUES DE ST. GOBAIN, CHAUNY & CIREY. Brit. 520,673, May 15, 1940 (May 14, 1938).

Internally frosted glass article and method of making. H. S. WILLSON (Corning Glass Works). U. S. 2,202,326, May 28, 1940 (April 26, 1938).

Lehr feeder. J. E. FIGEL (Fairmount Glass Works, Inc.). U. S. 2,203,385, June 4, 1940 (Dec. 3, 1938).

Machine for polishing or grinding lenses, etc. D. TAYLOR. Brit. 520,603, May 8, 1940 (Nov. 15, 1938).

Manufacture of sheet glass. A. J. BUNDY (American Window Glass Co.). U. S. 2,201,286, May 21, 1940 (May 4, 1938).

Method and apparatus for making glass. VERGIL MULHOLLAND (Hartford-Empire Co.). U. S. 2,203,269, June 4, 1940 (Feb. 14, 1938).

Method and apparatus for manufacturing products by subjecting a material to relatively high temperatures. LEE R. CAMPBELL. U. S. 2,029,307, Feb. 4, 1936.—A new type of cupola for melting raw material for mineral-wool production. J.S.

Method and apparatus for use in the manufacture of laminated safety glass. E. L. WALTERS (Libbey-Owens-Ford Glass Co.). U. S. 2,205,003, June 18, 1940 (July 14, 1938).

Mineral-wool insulation. GEORGE D. SHAVER. U. S. 2,101,921, Dec. 14, 1937.—Cylindrically shaped forms of mineral wool are formed by suction from a vat containing a wet mix of mineral wool and binder. J.S.

Ornamental glassware. A. J. CUNNINGHAM AND VICTOR WALKER (A. H. Sharpe). U. S. 2,203,647, June 4, 1940 (May 27, 1939).—A solid transparent glass stem for a beverage drinking glass comprises an element of polyhedronal geometric form having a transverse section in the form of a polygon with no directly opposed parallel sides.

Plastic for laminated safety glass. J. D. RYAN (Libbey-Owens-Ford Glass Co.). U. S. 2,205,020, June 18, 1940 (April 28, 1937).

Producing a cellular glass product. A. D. NASH (Pittsburgh Plate Glass Co.). U. S. 2,202,714, May 28, 1940 (Oct. 20, 1937).

Producing colorless transparent glasses. ALFRED THÜRMER. Ger. 686,383, Dec. 14, 1939 (Aug. 29, 1934); VI/32*b*. 2.—Colorless transparent glasses are produced by using the usual decolorizing agents such as selenium compounds, cerium salts, nickel oxide, cobalt oxide, manganese dioxide, etc. The decolorizing compound is added to the mass containing fluorine compounds, especially alkali fluorides, in an amount which would not bring about a complete decolorization without the presence of the fluorine compound. D.A.B.

Producing copper-ruby glasses. FRANK DOBROVOLNY, JOHN M. YOUEL, AND HAROLD KLEIN (E. I. du Pont de Nemours & Co.). Ger. 685,688, Nov. 30, 1939 (Dec. 6, 1938); VI/32*b*. 4.—Copper compounds are added to fused glass batches. Cyanogen compounds such as cyanides, cyanamides, or cyanates of alkali metals are added to the glass batch or glass melt. D.A.B.

Producing curled glass threads. ADOLF DIETZEL (Kaiser-Wilhelm-Institut für Silikatforschung). Ger. 683,867, Oct. 26, 1939 (Dec. 13, 1937); VI/32*a*. 25.—Curled glass threads are produced by bringing together glass fibers having different coefficients of expansion. The different kinds of glass are melted simultaneously in a multichambered receptacle whose walls are so arranged that the individual kinds of glass are kept separated until drawn from the spinning openings of the individual chambers. D.A.B.

Producing silicate wool objects having a vitreous-like coating. WILLY HOFFMANN (Günther Hoffmann and Diether Hoffmann). Ger. 682,181, Sept. 21, 1939 (May 7, 1938); VI/80*b*. 22.05.—Small objects are produced from silicate wool and a water-hardening binding agent. A plastic mass is formed and is heated until a vitreous coating is produced on the outer surface. D.A.B.

Safety glass. E. L. FIX (Pittsburgh Plate Glass Co.). U. S. 2,202,690, May 28, 1940 (July 7, 1938).

Shatterproof article of glass. H. R. OWEN (Electrical Products Corp.). U. S. 2,202,396, May 28, 1940 (July 1, 1938).

Tempering glass articles. W. W. SHAVER (Corning Glass Works). U. S. 2,205,180, June 18, 1940 (Sept. 24, 1935).—In a method of tempering glass, the steps comprise melting a salt which tends to become alkaline on decomposition to form a bath, maintaining the salt bath at a temperature at which it will tend to decompose, maintaining in suspension in the bath a finely divided substance which will remain solid at the temperature of the bath and will neutralize the alkalinity of the bath, uniformly heat-

ing the glass by immersing it in the bath, and suddenly chilling the glass.

Tempering glass articles, and bath therefor. W. W. SHAVER (Corning Glass Works). U. S. 2,205,181, June 18, 1940 (July 31, 1935); June 2, 1937).—(1) In a method of tempering glass, the steps comprise melting a salt which tends to become alkaline on decomposition to form a bath, maintaining the bath at a temperature at which it will tend to decompose, maintaining in the bath a small quantity of material which will neutralize the alkalinity of the bath, and immersing the glass in the bath. (5) A substantially anhydrous fluid treating bath for glassware comprises a molten salt of an alkali metal and a substance which will react with free alkali and prevent the bath from becoming alkaline.

Tempering hollow glass articles. JESSE LITTLETON AND RALPH DAY (Soc. Anon. des Manufactures des Glaces & Produits Chimiques de St. Gobain, Chauny & Cirey). Ger. 685,512, Nov. 30, 1939 (June 24, 1938); VI/32*a*. 30.—A process is given for tempering hollow glass articles whose form and wall strength vary at different spots. Hollow glass articles, especially flasks, are dipped into a cooling bath, a cooling liquid is introduced into the interior of the article, and the article is rotated. D.A.B.

Windowpanes having a higher elasticity of compression. GEORG JAECKEL. Ger. 682,346, Sept. 28, 1939 (Aug. 20, 1937); VI/32*a*. 20.—Windowpane glass is strengthened at regular intervals by ribbing; this ribbing constitutes the major portion of the glass and forms a large part of the thickness. D.A.B.

Writing surface. J. E. ARBERRY (Pittsburgh Plate Glass Co.). U. S. 2,202,682, May 28, 1940 (March 25, 1939).

Structural Clay Products

Black cores in brick. P. THOR. *Tonind.-Ztg.*, **62** [99] 1107–108 (1938).—Black cores in brick are caused by bituminous matter that is not completely burned out in the early stage of the firing before the surface vitrifies. They can be prevented by proper firing, even if the rate of firing has to be speeded up to increase production. W.K.

Causes of laminations on dearing. G. WELZEL. *Tonind.-Ztg.*, **62** [51] 554–56 (1938).—Laminations are due to the lack of uniformity of the body and to the effect of the auger. A high vacuum helps to decrease or to eliminate this trouble. W.K.

Disintegration of face brick by crystallization of soluble salt. J. M. HARDESTY. *Proc. Amer. Soc. Testing Materials*, **39**, 971–78 (1939).—H. describes a study carried out to determine the cause of the disintegration of the face brick in a building only a few years old. Selected samples were cut from the building, subjected to freezing and thawing tests, and examined chemically and microscopically for the presence of soluble salts. The facts determined indicate that (1) the observed weathering of the brick is due not to frost action but to the formation of crystals of calcium sulfate on and just underneath the exposed face of the brick and (2) the source of the calcium sulfate is in the body of the brick itself, and it occurs in a soluble form. R.A.H.

Firing of vitrified brick. A. MÖSER. *Tonind.-Ztg.*, **62** [65] 706–708 (1938).—The firing of vitrified brick is a difficult task. Salting and reducing must be done carefully. Downdraft kilns are the most suitable. W.K.

Impregnating cement mortar with coal pitch. M. J. CHIGEROVITSCH AND M. N. BRILLIANTOV. *Nauch.-Tekh. Inst. Prom. Stroitel. & Vyazhuschtschikh Material.*, pp. 57–79 (Jan., 1936); abstracted in *Referat. Silikatliteratur*, **6** [3] 6237 (1939).—A cement-sand mortar was mixed with 5 to 15% (dry weight of the mortar) of powdered coal pitch. The specimens were heated in an autoclave to 170° to 173° (8 atm. pressure). The results of a series of tests are as follows: (1) the addition of 5 to 15% of pitch had no effect on the consistency of the mortar; (2) with increasing additions of pitch, the beginning of setting was retarded, but the end of the setting was not changed; (3) microscopical examination of the specimens before the thermal treatment showed that a large part of the pitch settled around the sand grains; (4) after treatment in the autoclave, the sand grains were more tightly enveloped by the particles of pitch which became rounded; (5) after steaming, the specimens were less resistant to pressure, but resistance to breaking was greater; (6) with additions of up to 7.5% pitch the resistance to pressure decreased because of the thinning; with additions of more than 7.5% the resistance to pressure was reduced to such an extent that the pitch hindered the setting; (7) the resistance to breaking increased with additions of up to 7.5%; (8) with higher additions of pitch the capillary tension of water decreased; with 7.5 to 10% pitch, the capillary tension of water was reduced to zero in the heat-treated specimens; (9) this conditions a certain decrease in the water saturation, hygroscopicity, and permeability to water and an increase in the chemical resistivity; (10) the water saturation of the steamed mortar containing pitch decreases with higher pitch content (in spite of the increasing thickness of the mortar); (11) similar conclusions can be drawn with regard to the hygroscopicity; (12) the water permeability of the treated specimens is smaller than that of those not treated; (13) the resistance to aggressive waters is greater for mortar containing pitch. M.V.C.

Industrial production of keramsite. G. L. LAGUNOV AND N. D. RASTORGUEV. *Prom. Stroitel. Materialy*, 1939, No. 1, pp. 124–28.—The technological process of producing lightweight aggregates comprises the use of clays with a low Fe_2O_3 and CaO + MgO content. Coarse-grained quartz, pyrite, and gypsum admixtures have a negative effect. A slightly modified tunnel kiln is used; the firing process takes 9 hr. and 20 min., the highest temperature being 1200°. P.B. & E.S.

SEPARATE PUBLICATION

Fire Resistance of Building Materials. HARRY D. FOSTER. *Ohio State Univ. Eng. Expt. Sta. Bull.*, No. 104, 58 pp. (1940); *Ohio State Univ. Eng. Expt. Sta. News*, **11** [5] 11–13 (1939).—Standard fire tests were run on 31 walls made from hollow clay partition tile, various standard building materials in load-bearing walls (end and side construction fire-clay tile; gravel, cinder, and Haydite concrete; and some of these also with a brick facing), and partition walls of chemically bloated clay units especially designed for increased fire resistance. About twenty of the walls were 5 x 5 ft. rather than the standard 10 x 10 ft. size. The conclusions are that (1) thin unplastered partitions of shale or surface clay tile will not give satisfactory performance; (2) thin unplastered fire-clay tile are satisfactory if end construction is used; (3) unplastered 4 in. single cell partition walls of fire-clay tile give 30 min. fire resistance; 6-in. walls give a few minutes longer; (4) walls such as those in 3, plastered on both sides with ¾ in. gypsum plaster, give 60 to 75 min. fire resistance; (5) fire resistance of walls such as those in 3 will be 3 to 4 times as long if the cells are filled with a granulated insulating material; vesiculated slag was used; (6) thin partition walls of clay block made artificially with both 50 and 75% porosity give 3 to 4 times as much resistance as hollow clay tile of the same thickness; and (7) increasing the number of cells through the thickness of the block will materially increase fire resistance. Detailed descriptions are given of all tests, together with graphs and drawings. See *Ceram. Abs.*, **19** [7] 163 (1940). W.D.F.

PATENT

Brick-forming machine. A. F. STAVENOW. U. S. 2,203,447, June 4, 1940 (Oct. 6, 1938).

Refractories

Apparatus for measuring thermal conductivity of refractories. CESARE CODEGONE. *Ricerca Sci.*, **10** [7–8] 701–706 (1939).—An electrically heated furnace based on the auxiliary wall method and furnished with guard heaters to avoid radial temperature gradients has been used to measure the heat conductivity of refractories up to about 1000°C. V.S.M.

Chromite brick. C. KOEPPEL. *Tonind.-Ztg.*, **62** [59] 637–40 (1938).—Papers on chromite brick taken from K.'s book (*Ceram. Abs.*, **17** [9] 308 (1938)) are reviewed. W.K.

Effect of impurities on the sintering of magnesium oxide. GIOVANNI MALQUORI AND VITTORIO CIRILLI. *Ricerca Sci.*, **10** [10] 905–14 (1939).—Finely ground pure MgO was pressed into briquettes under a pressure of 350 kgm. per sq. cm. To obtain a thorough mixture of the impurities with the MgO, the briquettes were treated under vacuum with solutions containing salts of the impurities in a form that would not be affected by the alkalinity of the magnesia. Only the silica was added as silica gel to the powdered MgO. The treated briquettes were calcined between 700° and 900°C., after the addition of each impurity, and then sintered for 4 hr. at 1300°, 1450°, 1500°, and 1550°C. The porosity and the slaking of the sintered products were determined, and X-ray studies were made. The slaking tests were carried out by treating the briquettes sintered at the highest temperature in an autoclave under a pressure of 5 atm. for 30 min. and then drying them in a desiccator over P_2O_5 and KOH. The results are summarized in the following table:

Fe_2O_3 (%)	CaO (%)	SiO_2 (%)	Al_2O_3 (%)	Porosity of the material (%) at 1300°	1450°	1500°	1550°	Slaking increase in wt. (%)
				51.4	49.5			38.3
5.5				19.2	15.2			3.2
10.4				18.0	14.3			2.7
5.15	1.75			3.7	2.5			1.5
5.21	5.48			2.7	2.1			
		1.5		42.0		37.5		38.0
		5.0		51.0		47.0		39.0
	2.05	1.5		43.0		39.5		42.0
	1.95	5.0		49.0		42.5		19.0
2.87		1.5		35.5		34.5		27.2
2.95		5.0		44.0		43.0		36.0
2.37	2.05	1.5		34.0		33.8		11.0
2.39	1.95	5.0		41.5		41.0		26.0
			2.5	35.0			14.0	34.0
			7.5	39.3			16.0	26.0
			10.0	39.6			23.5	29.0
	1.4		2.5	16.0			2.0	0.4
	4.2		2.5	17.0			13.7	9.2

X-ray studies show that the presence of Fe_2O_3 and CaO increased the crystal size of the calcined MgO. V.S.M.

Fused diabase in reaction apparatus and some peculiarities of its use. L. YA. POPILOV. *Zhur. Khim. Prom.*, **15** [10] 16–23 (1938).—The difficulty of producing complicated shapes from diabase makes it necessary to construct the apparatus of several parts and bind them with a special putty. The latter is prepared from 92 to 94% of ground fused diabase, 4 to 5% of sodium silicofluoride, and 2 to 3% of barium silicate. The preparation and use of the putty are described in detail. Diabase apparatus for sulfite cellulose production is quite corrosion resistant, as no liquid enters into its body; the liquid can enter into the putty to some extent, but the formation of a solid superficial layer isolates it from the contents of the apparatus. P.B. & E.S.

Heater radiants: specialized process. R. M. RICHTER. *Ind. Gas*, **18** [11] 10, 18 (1940).—The Lloyd L. Sissell Co. manufactures heater radiants from ball clay, kaolin, grog, talc, lead carbonate, and sawdust. Radiants are fired in a circular gas kiln. S.S.

Improvement of the quality of standard refractory brick. A. MÖSER. *Tonind.-Ztg.*, **62** [18] 195–97; [20] 231–32 (1938).—M. discusses the possibilities of improving the preparation of the body and the process of forming refractory brick of standard dimensions, especially the deairing at the press. **Discussion.** F. GAREIS. *Ibid.*, [57] 615–16.—The deairing of finished pieces of large size and complicated shape in special vacuum chambers has a beneficial influence upon the resistance to thermal shock. **Reply.** A. MÖSER. *Ibid.*, [62] 668–69. W.K.

Introduction of solid fuel into bauxite charge in sintering. K. E. MANOĬLOV AND A. A. BENGLYANTS. *Tsvetnye Metally*, **1939**, No. 2, pp. 114–18.—Data are reported on the effect of the addition of coal, peat, and anthracite on the sintering of a charge of bauxite, limestone, and soda. The charge was ground to −120-mesh, and the fuel was added in amounts calculated to produce from 10 to 25% to 50% of the total heat required for sintering by the usual process. The charges were sintered in laboratory furnaces of the muffle type for 30 min. at a final temperature of 1200° to 1250°. The product was subjected to chemical analysis and the usual wet treatment in order to determine the soluble alumina. The results show that the use of solid fuel did not reduce the content of soluble alumina and alkali. The products had porosities of 20 to 55% as compared with 5 to 8% without additions of solid fuel. The degree of fuel combustion was satisfactory, the best results being obtained with anthracite. B.Z.K.

New refractory material, Magnite. SUNAO KORA. *Jour. Japan. Ceram. Assn.*, **48** [566] 68–73 (1940).—Magnite is the trade name of the basic refractory material which was developed and patented by Kurosaki Ceramic Co. Its main constituent is forsterite. K. discusses and reviews the development of forsterite refractories in the world and also describes the development of Magnite. Its properties are as follows: P.C.E., 37; true specific gravity, 3.2; bulk specific gravity, 2.5; chemical composition, SiO_2 30, Al_2O_3 2, Fe_2O_3 7, MgO 56%; thermal expansion, 1.02% at 1000°C.; thermal conductivity, lower than that of chrome or magnesite; and commencement of deformation under load of 2 kgm. per $cm.^2$, 1360°C. (that of ordinary magnesite brick is 1250°C.). Magnite is now used in many steel plants as a substitute for chrome brick or magnesite brick, and the results are very satisfactory. Crushed Magnite is used as metal encased brick and gives excellent results. Some steel plants are now testing the use of crushed Magnite as a stamping material in the open hearth. Y.K.

Preparation of alumina: I, Hydrolysis of aluminum acetate solution. SHOHEI UNO. *Waseda Applied Chem. Soc. Bull.*, **13** [27] pp. 1–9 (1936).—U. attempted to prepare alumina by the following procedure: (1) the aluminum sulfate was obtained from aluminum ore, (2) the crystalline precipitate of basic aluminum acetate was obtained by heating a mixed solution of aluminum sulfate and ammonium acetate in the pressure bottle, and (3) the basic aluminum acetate was then converted to aluminum oxide. As the first step in this procedure has been well investigated, U. made several experiments on the second and third steps. He studied the hydrolysis of the aluminum acetate solution. The results are summarized as follows: (1) the solutions of aluminum sulfate and ammonium acetate were mixed in various proportions and heated at 100° to 130°C. for 2 hr. in a pressure bottle; a high percentage (about 90%) of alumina precipitate can be obtained from the solutions, which contain over 0.2 mol. of ammonium acetate per liter; (2) by increasing free acetic acid, a higher percentage of alumina precipitate was obtained; (3) in studying the heating conditions, good results were obtained by heating at 100°C. for $^1/_2$ to 1 hr., and the percentage of basic aluminum sulfate in the precipitate has a tendency to increase when the solution is heated at a higher temperature; (4) in the cases where the higher precipitation percentage of alumina was obtained, the hydrolysis

product was basic aluminum acetate containing 4 mol. of acetate radical to 1 mol. of alumina; (5) basic aluminum acetate, thus obtained, was crystalline; it can be easily filtered and washed. For Part II see *Ceram. Abs.*, **16** [10] 304 (1937). Y.K.

Refractory material. E. BELANI. *Tek. Tid.*, **70** [2] Bergvetenskap 7–8 (1940).—B. reviews methods of making crucibles for melting pure metals and alloys. For melting pure iron, the use of either Greek magnesite prepared by the acetate method or electrically sintered magnesite is not recommended, but a mixture of Greek magnesite with 15% of cleaned and fired zirconium silicate makes an excellent crucible. With such crucibles, however, the danger of the iron taking up silicon and phosphorus must be guarded against; this is done by mixing finely ground magnesia, calcined at 1600°C., with 10% by weight of magnesium chloride ($MgCl_2 \cdot 6H_2O$) or with shellac dissolved in alcohol. The mixture is then rammed in a mold; on completion of the ramming, one half of the mold is removed and the crucible is allowed to dry in the other half; it is then fired at about 1600°C. Such crucibles can be used for melting iron and nickel, and a very high degree of purity is maintained. R.A.R.

Report of Committee C-8 on Refractories of the A.S.T.M.: Appendix I, Industrial survey of conditions surrounding refractory service in lime burning. *Proc. Amer. Soc. Testing Materials*, **39**, 345–48 (1939).—The manufacture of lime in two different types of kilns, viz., the vertical and the horizontal, is described. The furnace is divided into four zones, and recommendations are made relative to the quality of brick best suited for each zone to best withstand the particular type of service condition outlined. For **Appendix II** see *Ceram. Abs.*, **19** [7] 164–65 (1940). R.A.H.

Saving money in the boiler house. S. N. DUGUID. *Steam Engr.*, **9**, 172–74, 218–20 (1940).—The chemical and physical properties and tests of refractories for boiler furnaces and flues are explained simply, particular attention being paid to the use of Seger cones for the measurement of refractoriness. Other points dealt with include allowance for thermal expansion in a structure and the porosity, hardness, refractoriness under load, and aftercontraction of firebrick. L.R.B.

Slagging process of refractory brick. K. ENDELL. *Ber. Deut. Keram. Ges.*, **19** [12] 491–513 (1938).—The properties on which the sale of fire-clay brick is at present based, viz., alumina content and P.C.E., give no indications of the resistance to slag attack. The chemical composition also is of comparatively little importance. The important factor is low porosity accompanied by the resistance to abrupt changes in temperature. Problems which must be studied jointly by the manufacturers and users of refractories are (1) solubility (that quantity of unfused refractory materials which a slag can take up without solidifying partially or completely); (2) degree of fluidity of slags at high temperatures and attack on refractory brick; and (3) hydrodynamic conditions. A description of Fehling's method of determining the solubility graphically is given. Two types of viscometers (W. Hähnlein and G. Heidtkamp) for measuring viscosity at elevated temperatures are briefly described, and a short account is given of the apparatus for determining the rate of flow and the film thickness of liquids on inclined surfaces. It is desirable that the eutectic temperature of the reaction products between refractories and slags be higher than the working temperature, and this is most readily attained with chrome-magnesite brick. The marked increase in the rate of slag attack with rising temperature is due to the threefold effect of increased solubility, reduced viscosity, and accelerated diffusion. Most effective counter measures against slag attack are choice of refractories with minimum solubility, reduction of fluidity of the slag by the addition of insoluble substances, and cooling of the walls to a temperature about 50° above the melting point of the slag. The interrelationship of all the different factors is shown diagrammatically. See *Jour. Amer. Ceram. Soc.*, **22** [4] 105–16 (1939). E.J.V.

Soaking pit refractories: I. EVERETT C. HITE AND GILBERT SOLER. *Brick & Clay Record*, **96** [1] 62–63 (1940).—The three general types of soaking pits commercially in use at the present time are the regenerative, recuperative, and direct-fire types. In recent years considerable improvement has been made in the design and type of equipment used in soaking pits. Automatic control used on the newer furnaces not only has increased the efficiency of ingot heating but also has standardized conditions so that a more comprehensive study of the refractories used can be made. Normally, first-quality and in some cases superquality fire-clay brick are used for the roofs of soaking pits. These brick must have good spalling and abrasion resistance and good mechanical strength. Insulating brick have not proved entirely satisfactory in the suspended type of roof, largely because of low mechanical strength. Insulating brick are being used in the spring arch installations. Side walls are built of fire clay, "Yough" silica brick, or silica stone. These materials must resist spalling and slagging. Lower portions of the side walls, however, are usually built with chrome, chrome-magnesite, or magnesite brick. These are used because of their resistance to the high iron-oxide slag which is formed in the bottom of the pits. The bottom of pits usually consists of 13 to 18 in. of dense first-quality firebrick covered with 6 to 12 in. of coke breeze. The most difficult sections in a pit from a refractories standpoint are the port end arches. Generally, Yough or other silica brick and, in some cases, superduty firebrick or semisilica brick that have good resistance to load and spalling are used. Burners, burner ports, checkerwork, and insulation are briefly discussed. **II.** *Ibid.*, [2] 60–62.—The design of the recuperative soaking pit is briefly discussed. The roofs of this type of pit are generally built with either exceptionally high spalling resistant first-quality brick or superquality firebrick. Side walls are generally built with Yough silica brick or silica stone backed up with insulating brick. In the lower portions of the side wall a basic or neutral refractory is used in order to withstand the chemical attack of the high iron-oxide slag. The refractories used in the pit bottoms are similar to those used in the regenerative type of soaking pits. In the vertical type recuperators, first class air hardening fire-clay cement is used for setting all tile. The refractory materials used in this type are die-cast clay, silicon carbide, and electrocast mullite. The design of the direct fire soaking pit is briefly discussed, and the refractories used are similar to those used in the recuperative type of soaking pit. Six references are given. J.J.H.

Technical progress in refractories during 1939. ANON. *Brick & Clay Record*, **96** [1] 64–65 (1940).—The methods of preparation, materials, research and testing, and the application of various refractories are reviewed. 20 references. J.J.H.

X-ray study of natural and artificial mullites. H. P ROOKSBY AND J. H. PARTRIDGE. *Jour. Soc. Glass Tech.*, **23** [100] 338–46 (1939).—X-ray studies indicate that there are three varieties of mullite distinguishable by the difference in axial ratios of the unit cell. They are α-mullite, made from pure materials corresponding to the formula $3Al_2O_3 \cdot 2SiO_2$ and containing 28.2% SiO_2 and 71.8% Al_2O_3; β-mullite, containing excess alumina in solution; and γ-mullite, containing small proportions of ferric oxide and titania in solution. All three varieties have been prepared artificially, but so far only the β and γ varieties have been found as minerals. β-mullite can contain as high as 78% of alumina, although there is a progressive change in the lattice from the α to the β variety as the alumina content rises above 72%. γ-mullite is obtained when comparatively small proportions of iron or titanium oxides are present; with larger proportions, a general expansion of the lattice occurs. β-mullite containing 78% alumina corresponds to the formula $2Al_2O_3 \cdot SiO_2$. It is clear that the replacement of silicon by aluminum is not limited to the change from sillimanite to α-mullite but can be carried a stage further until the composition of β-mullite is reached. G.R.S.

PATENTS

Brick for regenerators. BONNYBRIDGE SILICA & FIRECLAY CO., LTD., AND W. MCBRYDE. Brit. 521,024, May 22, 1940 (Nov. 10, 1938).

Cast refractory block and method of producing. G. S. FULCHER (Corhart Refractories Co.). U. S. 2,201,723, May 21, 1940 (Sept. 28, 1937).

Containers or pots for molten baths in the heat treatment of metals. G. SIEBERT GES. Brit. 520,670, May 15, 1940 (March 12, 1938).

Heat exchanger made of impervious sintered ceramic material. WERNER RATH (Porzellanfabrik Kahla). Ger. 685,246, Nov. 23, 1939 (Nov. 20, 1937); VI/80*b*. 8.08.—A ceramic mixture consisting of magnesium silicate (rendered impervious upon firing) is used. The magnesium oxide content exceeds the proportion of 40 parts MgO to 60 parts SiO_2; the preferable proportion of MgO to SiO_2 in the mixture is 40:55 to 60:45 parts, and the desirable clay material is present to the amount of 15% aluminum oxide. Other admixtures favoring thermal conductivity are added, such as titanium dioxide, silicon carbide, corundum, or mixtures of these materials. D.A.B.

Manufacture of burner elements. V. W. KLIESRATH (International Engineering Corp.). U. S. 2,201,777, May 21, 1940 (Jan. 6, 1938).—The method of making perforated refractory articles comprises forming a paste of refractory material, placing the paste in a mold having series of oppositely disposed pins, pairs of which are aligned with each other and with their ends spaced apart to leave a web substantially central of the material, drying the material in the mold, separating the mold in the plane of the web, removing the material from the mold and firing it, and thereafter punching out the material of the web between the aligned sockets formed by each pair of pins.

Manufacture of insulation material. JOHN C. KERSHAW (Eagle-Picher Lead Co.). U. S. 2,130,091, Sept. 13, 1938.—A block of high-temperature insulating material is made by molding asbestos fibers, exfoliated vermiculite, and magnesium oxysulfate crystals. The molded material is heated for 48 hr. at a temperature not higher than 300°F. J.S.

Mold composition and method of producing. V. A. GRODSKY. U. S. 2,201,366, May 21, 1940 (Aug. 2, 1939).—A mold composition comprises graphite particles, dehydrated clay particles, and a binder adapted to unite the graphite particles with the clay particles when the composition is subjected to that degree of heat sufficient to dehydrate the clay particles.

Producing refractory crucibles. CLARENCE J. BROCKBANK AND SOC. D'ÉLECTRO-CHIMIE, D'ÉLECTRO-MÉTALLURGIE ET DES ACIÉRIES ÉLECTRIQUES D'UGINE. Ger. 683,676, Oct. 19, 1939 (April 18, 1936); VI/80*b*. 8.11.—Refractory crucibles of high mechanical resistivity are produced from silicon carbide, graphite, and sand and/or an organic binding agent. High-melting metals such as iron, nickel, chromium, manganese, or vanadium are added to the ground mass containing natural graphite in flakes, and the mass is fired at a low temperature of 800° to 900°C. in a reducing atmosphere. D.A.B.

Refractory block. V. M. GOLDSCHMIDT. Ger. 682,062, Sept. 14, 1939 (June 28, 1938); VI/80*b*. 8.18.—This refractory block consists of a charge having a metallic covering of iron or steel. The filling of the block has a charge high in magnesium orthosilicate content, such as olivine stone. D.A.B.

Refractory covering for metallurgical furnaces. ERNST JUSTUS KOHLMEYER. Ger. 681,315, Aug. 31, 1939 (July 25, 1936); VI/80*b*. 8.09.—A furnace covering is made of a mixture of zinc oxide with other refractory oxides such as aluminum oxide, chromium oxide, chrome iron ore, or zirconium oxide. D.A.B.

Refractory and method of making. R. C. BENNER AND G. J. EASTER (Carborundum Co.). U. S. 2,203,770, June 11, 1940 (Oct. 25, 1937).—A glass tank comprises cast refractory blocks consisting principally of alumina and containing over 5% of iron oxide.

Spalling-resistant refractories. A. W. PARFITT (Honorary Advisory Council for Scientific & Industrial Research). Brit. 521,055, May 22, 1940 (Nov. 12, 1938).

Terra Cotta

Chemical contribution to the study of antique terra cotta. S. JACCARINO. *Ann. Chim. Applicata*, **30**, 132–41 (March, 1940).—J. reports the chemical constitution of 21 fragments of terra cotta of varying origin and epochs. K.R.

Control of soluble metallic salts results in uniform colors. JOHN H. GRADY. *Brick & Clay Record*, **96** [2] 31 (1940).—The advantages of metallic soluble salts over colored glazes for polychrome decorations are (1) ease of application, (2) production of many desirable colors and intensities not otherwise possible, (3) cheapness, (4) better grade of product, and (5) applicability over monochrome or mottled textures. A base glaze of monochrome or mottled texture is applied to the body and permitted to air dry, and the soluble-salt solutions are applied to the glaze by either painting or spraying. Aqueous solutions of the majority of these soluble salts have a hydrogen-ion concentration lower than pH 7.0, and therefore metal parts cannot be used in painting or spraying. Experiments conducted with copper chloride, copper nitrate, cobalt chloride, and chromium nitrate are described. J.J.H.

Improving rural conditions and needs of livestock on farms. ANON. *Ceramica*, **2** [3] 96–99 (1940).—Fire-clay or stoneware feeding and watering troughs for poultry and livestock are resistant to acids and are more durable than cement or wood feeding troughs. Stoneware equipment for barns, pigsties, and poultry yards is now manufactured. Illustrations are from the Pozzi Mfg. Co., Italy. M.V.C.

PATENTS

Ceramic article. P. C. KINGSBURY (General Ceramics Co.). U. S. 2,201,684, May 21, 1940 (Sept. 2, 1937).—A ceramic vessel has a silicon carbide body portion, an annular ring of stoneware bonded thereto, the outer surface of which is ground to present a smooth contacting surface, and a lid of ceramic material engaging the smooth stoneware surface for tightly closing the vessel.

Engobing and dipping plant for ceramic articles. WILHELM KÜMMEL. Ger. 685,238, Nov. 23, 1939 (June 30, 1938); VI/80*a*. 58.—An engobing and dipping plant for ceramic articles has a continuously revolving rocking feeder which dips the articles to be engobed into a receptacle filled with engobing material. The shaker of the feeder operates by the swinging of a double-armed lever which is motivated by contacts on a motor. The feed chains run over sprocket wheels and allow the lowering and lifting of the article into the dipping receptacle at intervals corresponding to the swinging and shaking motions of the receptacle. D.A.B.

Flowerpot for water-cultured plants. W. P. MUNSELL. U. S. 2,205,303, June 18, 1940 (June 9, 1938; renewed May 3, 1940).

Making interlocking roofing tile. DAVIS BROWN, J. B. PEEBLES, AND B. M. BURCHFIEL (Davis Brown). U. S. 2,205,080, June 18, 1940 (Aug. 4, 1936; Jan. 31, 1938).

Whiteware

Automatic jigger machines. ANON. *Ceram. Ind.*, **34** [6] 30–34 (1940).—A battery of three twin-unit automatic jiggering machines is used at the Homer Laughlin China Co. for forming certain standard flatware shapes at high speeds, averaging 20 plates per min. per unit, or about 172,800 pieces per day for the installation. Clay is furnished to the machines by horizontal steel tubes from the floor above the machine room. Clay is delivered under a hydraulic head of 1200 lb. per sq. in., accurately controlled by a series of pumps. Internal pressure in the clay column varies from 500 to 700 lb. per sq. in., depending on the plasticity of the clay. Clay batts are cut by a horizontally swinging cutting wire, synchronized to cut off just the right weight of clay and controllable within 0.5%. The central part of each automatic unit is a circular turntable, about 30 in. in diameter, carrying 6 alloy cast arms which pick up empty plaster molds from vertically moving conveyers. While arm 1 is receiving an empty mold, arm 2 is directly beneath one of the two clay delivery tubes, arm 3 is under the alternate delivery tube, arm 4 is forcing the clay batt against a brass mold for preliminary shaping, arm 5 is having its piece shaped by a rotating jigger tool, and arm 6 is delivering its formed piece to the conveyer for its trip to the drier. The brass preforming mold is steam heated and spaced just the proper distance from the plaster mold to force the batt to fill the gap completely. The jiggering operation is done by a rotating tool lubricated by a water spray, while the mold remains stationary. All excess clay is washed out by a high-pressure water jet and collected in a rough agitator from which it is reclaimed. After the ware is formed, the molds are automatically put back onto rings of the drier conveyer chain and carried through a 70- x 100-ft. drier. After drying, the ware is removed by strippers who take it off without removing the molds from their conveyer rings. Each stripper removes ware with one hand and stacks it in bungs of 12 on a shelf conveyer which moves slowly past. Mechanized jiggering has greatly increased output and prolonged mold life and produces more accurately shaped ware with less firing warpage. Illustrated. L.M.C.

Electrical and mechanical tests on long rod porcelain insulators. F. OBENAUS. *Hescho-Mitt.*, No. 78–79, pp. 1–46 (1938); abstracted in *Referat. Silikatliteratur*, **6** [3] 6291 (1939).—New features of the long rod insulator are the great length of the insulating body and its resistance to mechanical stresses. It is an important development for insulation in high tension free transmission and is an outgrowth of the "full core" insulator with fewer metal armatures. Tests on preliminary discharge showed that with long rod insulators, with and without guard armature, only discharge phenomena occurring on both electrodes are present and sparks pass freely between the armatures. Other tests showed that a given spark distance is used in rod insulators in the same way as in previous types. The long rod insulators produce no audible disturbing discharge in the receiver. The only danger is from electrical arcs which not only heat up the ceramic parts but also, by exceeding the elasticity limits of the porcelain, produce cracks and fissures; protection may be afforded, however, by special armature. M.V.C.

High-frequency trimmers from long rod insulators. ANON. *Hescho-Mitt.*, No. 78–79, pp. 53–56 (1938); abstracted in *Referat. Silikatliteratur*, **6** [3] 6292 (1939).—Long rod insulators have made possible the manufacture of trimmers for high tension in an entirely new way. The special design does away with the disadvantages of other types of trimmers, and the form with three supports is very resistant mechanically. These trimmers are more impervious to alternating current and impact overvoltage than other trimmers, and they prevent overcharging in the insulated iron parts and foot. Long rod insulators with a diameter of 60, 75, or 85 mm. are used in the new Hescho trimmers, according to requirements for mechanical strength, and they can easily be replaced. M.V.C.

Insulators. J. S. FORREST. *Elec. Rev.*, **126** [3257] 473–74 (1940).—F. discusses the English developments of the last five years in the porcelain-insulator field. Eight dimensioned drawings of high-voltage insulators and two curves of potential distribution with normal and stabilized insulators are presented. J.L.G.

New quartz raw materials for the whiteware industry: II. HEINZ MIELDS AND HANS LEHMANN. *Ber. Deut. Keram. Ges.*, **20** [3] 113–30 (1939).—Special examinations of various kinds of well convertible sands are described. Coarse Börtewitzer sand and Seok sand are compared with pure β-quartz materials, crystal sand from Hohenbocka, and Danish flint. The best grade of conversion had to be found with the smallest expenditure for milling and firing costs. The type of mineralizer most useful for the production and the quantities of its addition to avoid the production of an inferior quality and to improve the conversion to β-cristobalite had to be determined. Conversion was observed with various grinding periods. Three 1-kgm. batches of crystal sand of Hohenbocka, coarse Börtewitzer sand, and Seok sand were ground for 24, 72, and 144 hr. The results are tabulated. Wet milling for 72 hr. gave the best results. The sands were fired in a continuous kiln at Seger cone 6 and at cones 9–10. In all experiments the materials were unground. Other materials ground for 24, 72, and 144 hr. were fired at cones 6 and 9–10. X-ray examinations prove that only finely ground sands show β-cristobalite interferences. Results of X-ray examinations are tabulated, and the percentage content of β-cristobalite is calculated. Crystal sand of Hohenbocka, Börtewitzer sand, and Danish flint were fired for 3, 12, and 18 hr. at 1200° and 1290°C. The sand was ground for 72 hr., and the flint was crushed. The density of the fired trials was determined. From these results, the content of cristobalite was calculated. The results are tabulated. With long firing periods at a temperature of 1200° and 1290°C., crystal sand of Hohenbocka and coarse Börtewitzer sand show a heavy reduction of density at 1200°C. Cristobalite formation sets in at this temperature approximately linearly with the firing time. An optimum is obtained after firing for 3 hr. at 1290°C. Chalk ($CaCO_3$) was used as mineralizer. It improves the convertibility. Coarse Börtewitzer sand is superior. Special sands containing a high percentage of silicic acid are considered the most favorable raw material for whiteware batches. Various tests with Börtewitzer sand are described. X-ray results are given. Further examinations in regard to hair-tearing, humidity expansion, and density were made, and the results are tabulated. Dump sands and buddled sand, which are by-products of buddled kaolin, were examined with regard to their practical use in the ceramic field. Some dump sands are of sufficient purity to be used in china and whiteware mixes. They are free from troublesome inclusions, and iron and titanium contents are limited to a comparatively low percentage. Danish flint is much superior to pure quartz sand because of the temperature used in the earthenware industry. A lower firing temperature can be used with dump sands, and great savings are obtained in grinding time, current, mill material, fuel, and the life of furnaces. For Part I see *Ceram. Abs.*, **18** [8] 217 (1939). W.D.K.

Relations between English and Continental porcelain. W. B. HONEY. *Trans. English Ceram. Circle*, **2** [7] 88–98 (1939).—The rediscovery by Böttger of the art of porcelain making and the foundation of the Meissen factory are described, together with the subsequent spread of the secret. In 1750 Joseph Jacob Ringler learned how the kilns were made (the essential secret), and in rapid succession eight other factories were set up in Germany. These depended on Meissen style just as the English factories. In 1756, as a result of the Seven Years' War, the Meissen factory was eclipsed by Sèvres. English style changed in sympathy. Chelsea, Derby, and Staffordshire acquired their knowledge of soft-paste porcelain from France. Variants in England

were the addition of bone ash (Frye at Bow) and soapstone (probably Cookworthy). The English factories show their dependence on Meissen up to 1756 by their fondness for copying silverware and for flower and fruit painting; figures were also copied. After 1756, Sèvres colors, such as *gros-bleu*, were copied by Chelsea. Later, English factories were inventive, using the Sèvres elements. The dependence of English porcelain on that of the Continent waned. English porcelain had little influence on Continental productions. Though the modes were devised by Meissen and later by Sèvres, the taste and feeling expended by English factories within these modes were inferior only to these two among all the European factories. L.R.B.

Results of experiments on the effect of moisture on densely fired and porous porcelain by testing in the high frequency condenser field. W. Endres. *Hescho-Mitt.*, No. 78–79, pp. 47–52 (1938); abstracted in *Referat. Silikatliteratur*, **6** [3] 6290 (1939).—The difference between dense and porous fired porcelain was studied on dense and porous cylindrical porcelain disks of the same size. Tests were made on the specimens when dry and when soaked with water by subjecting them to high-frequency tension in the high frequency alternating field. The tests showed that a dielectric of dry porous porcelain does not get as warm as densely fired porcelain because of the greater amount of gas contained, i.e., the dielectric loss factor is lower in the case of porous porcelain. When the pores are filled with water, however, the loss factor can be more than the tenth power greater than that of densely fired porcelain, depending on the water content and the degree of porosity. The difference between dense and porous porcelain soaked in water is shown by their rise in temperature when exposed to high-frequency alternating tension. It is apparent even when a very small amount of water is absorbed. Besides increasing the loss factor, the absorption of water brings about another change in the dielectric constants and increases the heat. The results given show that subjecting high-tension insulators to high-frequency tension is a new method of testing porosity. M.V.C.

PATENTS

Ceramic support. A. L. Davis. U. S. 2,201,989, May 28, 1940 (Dec. 6, 1937).—In a vertically aligned assembly of a plurality of superposed fired ceramic ware support elements and a plurality of unfired ceramic articles in which each article is between two support elements, a ceramic ware support element comprises a member having a continuous outer portion of uniform thickness and substantially uniform width defining a space of greater extent than that occupied by the ceramic article and an inwardly declined continuous portion integral therewith for supporting the reverse of a superposed unfired ceramic article.

Covering for encased arc-light fusing electrode to be employed in weldings where special strength and toughness are necessary. Arnold M. Flixton (Allgemeine Elektricitäts-Ges.). Ger. 681,455, Aug. 31, 1939 (Jan. 8, 1937); VIIIc/21*h*. 30.15.—The covering for this electrode consists of 25 to 60% acid clay and 75 to 40% manganese dioxide. D.A.B.

Defecator with a combined flushing jet and air break. W. C. Groeniger (John B. Pierce Foundation). U. S. 2,202,628, May 28, 1940 (Sept. 11, 1937).

Movable core spark plug. Benjamin Miller (Power Patents Co.). U. S. 2,202,220, May 28, 1940 (April 10, 1939).

Producing condenser dielectrics from ceramic material. Steatit-Magnesia A.-G. Ger. 680,204, Aug. 3, 1939 (Jan. 6, 1936); VI/80*b*. 8.13. Addition to Ger. 676,263.—Condenser dielectrics are produced from an impervious sintered mixture of titanium dioxide and zirconium dioxide with magnesia as an alkali-earth admixing material according to Ger. 676,263. The magnesia and zirconium dioxide are introduced in part or completely into the mixture in the form of sintered zirconate. D.A.B.

Saggers and supports for use therewith in the firing of pottery. L. Bailly. Brit. 520,614, May 8, 1940 (Nov. 16, 1938).

Spark plug. William Harper, Jr. (H. B. Motor Corp.). U. S. 2,205,196, June 18, 1940 (June 16, 1937).

Spark-plug construction. A. L. Robb (R & R Mfg. Co.). U. S. 2,202,027, May 28, 1940 (Dec. 5, 1938).

Wear-resisting ceramic composition. W. B. Donahue (Feldspathic Research Corp.). U. S. 2,204,582, June 18, 1940 (Sept. 1, 1937).—A nonporous wear-resisting ceramic composition consists of unfused zircon bonded by a glass bond.

Equipment and Apparatus

Accurate direct-reading manometer for corrosive and other gases. R. Spence. *Trans. Faraday Soc.*, **36** [2] 417–19 (1940).—S. describes a glass manometer in which the relation between scale reading and pressure is linear. The dead space is small and practically constant (variation 1.25% for 1 atm.), and pressures up to 1 atm. can be readily determined to within 0.25 mm. Hg. The instrument is specially suited for the study of gas reactions. G.R.S.

Air heaters in brick plants. K. Spingler. *Tonind.-Ztg.*, **62** [57] 613–15 (1938).—The proper installation of steam heaters and of heat transferrers for the waste heat of the kilns to the driers is discussed. W.K.

Clay purification. J. Grewe. *Tonind.-Ztg.*, **62** [36] 403–405 (1938).—The latest types of clay washers are described. W.K.

Colloidal carbon as a grinding aid in Portland-cement manufacture. C. W. Sweitzer and A. E. Craig. *Ind. Eng. Chem.*, **32** [6] 751–56 (1940).—Grinding aids are becoming increasingly important in the manufacture of Portland cement because of their ability to improve materially the efficiency of clinker grinding. The experimental evidence indicates that colloidal carbon, commonly known as carbon black, is an effective grinding aid. A carbon dosage as low as 0.32% on the clinker increases the fineness of the cement 30% when the grinding time is constant; the same carbon dosage decreases the grinding time 28% when the grinding is run to constant fineness. These improvements are enhanced by increasing the carbon dosage. In terms of power saving and increased output, these results seem to be of practical significance. The cements prepared with carbon present as a grinding aid compared with the controls show improved strength properties in tensile and compression tests on mortars. The use of carbon in dosages up to 1% does not alter appreciably the standard properties of cement, with the exception of color, and has no noticeable effect on the resistance of cement mortars to freezing and thawing treatment. Illustrated. F.G.H.

Crushing operations in cement manufacture. Anon. *Concrete, Cement Mill Ed.*, **48**, 166–67 (1940).—Crushing uses 80 to 85% of the power in the plant; any reduction, therefore, is important. A concave crushing plate uses less power. A new type of hammer mill is reversible. A new gyratory crusher operates at a higher speed, and the spider and top shell are cast in one piece. A new plant in Texas using shell does not need primary crushing; the shell goes directly to a ball mill which is in closed circuit with a vibratory screen. W.D.F.

Deleterious coatings of the media in dry ball-mill grinding. Fred C. Bond and Fred T. Agthe. *Amer. Inst. Mining Met. Engrs. Tech. Pub.*, No. 1160; *Mining Tech.*, **4** [2] 10 pp. (1940).—When some materials are ground dry in a ball mill, a coating begins to adhere to the balls and the mill lining. The formation of this coating marks the practical limit of size reduction. Generally, the softer materials tend to coat at coarser sizes than the hard materials. Coating may be caused by (*a*) static electricity, (*b*) adsorption, and (*c*) mechanical impact; in *a* any charge on the balls is immediately conducted away and the particles are similarly charged, producing a repulsion;

in *b* an air film adsorbed on the particle tends to prevent contact with others; in *c* the particles are thought to be rammed into the scratches on the balls. This is supported by the laminated, concentric structure, increasing in density as the surface of the ball is approached. In the fine grinding of cement clinker, coating is a major problem. Laboratory tests of clinker plus 3% gypsum gave a strongly adherent coating. Microscopic examination showed pits on the balls with very fine particles wedged in them, slightly larger particles outside, etc. A test without gypsum showed less coating. When a very fine material, 3% ZnO, was added, coating was the worst observed. The addition of 0.88% water gave only a slight coating, as the very fine particles were hydrated and agglomerated. The addition of 0.75% water and 0.13% of a patented lignin compound gave almost no coating and the finest grinding observed in this series of experiments. W.D.F.

Determining the maximum dimensions of pores in filters. H. KNÖLL. *Kolloid-Z.*, **90** [2] 189–94 (1940).—The so-called bubble-pressure method for the determination of the pore size in filters, formerly described by H. Bechthold, M. Cantor, and R. Schurmann, is discussed. Bechthold's formula is as follows:

$$D = \frac{4 \cdot a \cdot 760 \cdot 10^4}{P \cdot 1.033 \cdot 10^3},$$

where D = diameter in μ, a = capillarity constant in dynes per cm., and P = bubble-pressure value in mm. Hg. K. shows that the values obtained by this formula are only approximate, as the rate of applied pressure is not taken into account, and that the bubble-pressure values and calculated pore diameters are the same for a single capillary lumen as for the maximum pores of bacteria filters. There is also no basis for Schurmann's assumption that the gas bubbles formed from fine filters during the pressure test originate from the union of microscopic bubbles rising from adjacent pores at the same time. Carbon tetrachloride was used as the test liquid. L.E.T.

Economics of air separation: I. J. HEYD. *Tonind.-Ztg.*, **62** [10] 107–109; [11] 119–20 (1938).—H. presents theoretical considerations on the efficiency of air separators and formulas for its calculation. **II.** *Ibid.*, [55] 593–95; [56] 604–607; [59] 640–41 (1938).—Results of practical tests on air separators are given and discussed. W.K.

Measuring open-hearth bath temperatures. L. O. SORDAHL AND R. B. SOSMAN. *Instruments*, **13** [5] 127–30 (1940).—The following methods of measuring open-hearth bath temperatures are described and compared: (1) dissolving-tube method, using a platinum thermocouple encased in porcelain and graphite, (2) test-spoon method, (3) bath-equalization method of Larsen and Shenk, (4) rod-boil method, (5) slag-bubble method, (6) slag-surface method, and (7) Collins-Oseland tube method. The Collins-Oseland tube held the most promise of becoming a practical and dependable method for routine use, although quite reliable readings were also obtained with the test spoon when the procedure was carefully standardized. J.J.H.

Physics and electrical insulation. A. M. THOMAS AND S. WHITEHEAD. *Jour. Sci. Instruments*, **17** [4] 77–83 (1940).—The theory of electrical conduction in gases, liquids, and solids is considered, and dielectric losses and dielectric constant are discussed. 35 references. J.L.G.

Quartz crystals. Their piezoelectric properties and use in the control of high frequency: I, History, theory, application, and performance. C. F. BALDWIN. *Gen. Elec. Rev.*, **43** [5] 188–94 (1940).—The so-called piezoelectric effect, i.e., "a pressure applied to certain substances in a direction parallel to one of the natural crystallographic axes; equal and opposite electric charges developed on opposite faces," was discovered by the Curies about 1880 during their investigation of pyroelectric phenomena. The piezoelectric function of quartz is used in conjunction with the vacuum-tube oscillator circuit to hold the frequency of the emitted signals or carriers within specified limits. L.E.T.

Recording water-flow meter. E. C. CHILDS. *Jour. Sci. Instruments*, **17** [4] 93–94 (1940).—C. describes the construction of a recording meter for measuring the instantaneous rate of flow of water from outfall pipes of drainage systems. Two constructed thus far have covered rates of flow up to 40 liters per min. Illustrated. J.L.G.

Resurfacing worn iron molds by the use of the Schoop electrospray pistol. M. U. SCHOOP. *Jour. Soc. Glass Tech.*, **24** [101] 58–59T (1940).—The surface layers of the cast-mold halves are cut away in order to obtain a better union and a larger surface and are then exposed to the discharge from the electrospray pistol, using iron or steel, for a sufficient length of time to obtain a deposit 6 to 8 mm. in thickness. The physical and chemical properties of the metal to be deposited should agree as closely as possible with those of the basic mold material. Essentially, the process consists in melting the metal to be deposited with an electric arc, so that the metallic particles are extremely hot and fluid when they come into contact with the surface of the mold and can thus form a close weld. Illustrated. See "Use . . .," *Ceram. Abs.*, **19** [1] 11 (1940). G.R.S.

Selecting a crusher. WILBUR GORMAN. *Mining Congr. Jour.*, **26** [4] 68 (1940).—Gyratory crushers have a greater capacity for reducing material to a given size than jaw crushers of equal receiving openings. Jaw crushers are usually used as primary crushers as they have a larger receiving opening for the same capacity. With a coarse feed, a straight jaw plate should be used; with only a little coarse feed, nonchoking surfaces should be used; they wear more uniformly. The Blake type jaw crusher has a larger capacity and the most leverage on the largest rock. The Dodge type is the reverse of this, but produces a more uniformly sized product. The single toggle produces a downward thrust on the material and has a large capacity. W.D.F.

Simplest ultracentrifuge with photographic recording. JAMES W. MCBAIN AND ALLEN H. LEWIS. *Trans. Faraday Soc.*, **36** [2] 381–84 (1940).—Details of the construction and operation of the simplest air-driven ultracentrifuge are given. The results are practically as good as those hitherto attained with any of the standard ultracentrifuges. See "Opaque . . .," *Ceram. Abs.*, **19** [1] 21 (1940). G.R.S.

Specific surface and particle-size distribution of finely divided materials. M. N. STATES. *Proc. Amer. Soc. Testing Materials*, **39**, 795–807 (1939).—General equations are given for specific surface and particle-size distribution of finely divided materials. Through the use of these equations the L. A. Wagner method (*ibid.*, **33** (Part II), 553 (1933)) may be applied to fineness studies in which various particle size ranges are used. An instrument and accessories are described which lend themselves to the use of a simplified procedure for fineness measurements. The unavoidable error in a single measurement of specific surface, using the Wagner procedure with any instrument, is probably 2%. In this method in which various particle size ranges are used, precise measurements of specific surface and particle-size distribution of powdered materials may be made with less than 5% of the material required for measurement with the Wagner turbidimeter. R.A.H.

Tension specimen shape and apparent strength. F. O. ANDEREGG, ROYAL WELLER, AND B. FRIED. *Proc. Amer. Soc. Testing Materials*, **39**, 1261–69 (1939).—The true maximum stress in a tension specimen is $K \times$ load/area, where K is a correction due to the nonuniformity of stress over the failure section. When the points of load application are well removed from the failure section, K is approximately $1 + d/4r$, where d is the width of the section at the neck and r is the radius of curvature. Fairly long, slender test specimens are thus indicated. R.A.H.

Use of the glass electrode at temperatures up to 100°C. R. G. HUMPHREYS. *Chem. & Ind.*, **58** [13] 281–82 (1939).—For a glass-electrode system to be of practical use, the

results obtained by it must be readily reproducible and calibration must be simple and rapid. A device is described and illustrated which achieves these two results at temperatures up to 100°C. The following factors are important: (1) the composition of the liquid within the electrode must remain constant; (2) liquid junction potentials must be constant; (3) the life of the bulb must be as long as possible; (4) contamination of the material under examination by diffusion must be avoided; (5) provision for rapid calibration at the desired temperature must be made; and (6) insulation must be adequate. The glass electrode differs from the hydrogen electrode in both the acid and the alkaline ranges, especially above 70°C., so that calibration curves must be used. When an apparatus is freshly set up, considerable difficulty arises in getting a steady calibration. After the apparatus is maintained for 48 hr. at 100°C., no further trouble occurs at any temperature. During the initial period the resistance of the bulb falls continually, e.g., from about 1 megohm to 80,000 ohms at 100°C. This is probably due to the gradual elimination of gas films and also to hydration of the glass. G.R.S.

Vibratory equipment. F. J. Van Antwerpen. *Ind. Eng. Chem.*, **32** [6] 765–70 (1940).—The applications of vibratory equipment in the operation of feeders, packers, and screens are described in detail. Illustrated. F.G.H.

SEPARATE PUBLICATION

Permissible Electrically Operated Pumps. L. C. Ilsley, E. J. Gleim, and H. B. Brunot. *U. S. Bur. Mines Repts. Investigations*, No. 3497, 23 pp. Free.—Information is given on the safety features of 24 mine pumps that have been approved as permissible by the Bureau of Mines before Oct. 1, 1939. R.A.H.

PATENTS

Abrasive blast machine. E. R. Hughes (American Foundry Equipment Co.). U. S. 2,204,596, June 18, 1940 (April 26, 1938).

Abrasive equipment. V. E. Minich and G. N. Grove (American Foundry Equipment Co.). U. S. 2,204,610, June 18, 1940 (Dec. 30, 1933). U. S. 2,204,611, June 18, 1940 (Dec. 30, 1933; May 5, 1939).

Abrasive-projecting machine. C. E. Unger (American Foundry Equipment Co.). U. S. 2,204,637, June 18, 1940 (June 9, 1938).

Abrasive-throwing machine. D. C. Turnbull (American Foundry Equipment Co.). U. S. 2,204,632, June 18, 1940 (Feb. 5, 1934).

Abrasive-throwing wheel. D. C. Turnbull (American Foundry Equipment Co.). U. S. 2,204,633, June 18, 1940 (Nov. 17, 1936). U. S. 2,204,634, June 18, 1940 (Nov. 17, 1936; Feb. 9, 1937).

Apparatus for:

Abrasively treating metal articles. R. W. Guite (American Foundry Equipment Co.). U. S. 2,204,588, June 18, 1940 (July 20, 1937).

Allaying dust from rock drills. J. T. McIntyre. U. S. 2,201,270, May 21, 1940 (April 10, 1937).

Making roofing granules. C. L. Colbert (Fiske Brick & Granule Co.). U. S. 2,202,090, May 28, 1940 (May 27, 1937; June 16, 1938).

Removing dust and other impurities from air and other gases. J. Yuille (Metal Wools), Ltd., and G. D. Baker. Brit. 521,061, May 22, 1940 (Nov. 12, 1938).

Treating metal articles. D. C. Turnbull (American Foundry Equipment Co.). U. S. 2,204,636, June 18, 1940 (Sept. 24, 1938).

Treating pulverulent materials. Nikolai Ahlmann (F. L. Smidth & Co.). U. S. 2,201,552, May 21, 1940 (May 15, 1936).—An apparatus for cooling pulverulent material, such as freshly ground cement, freshly hydrated lime, alumina, etc., comprises a container having a heat-conductive wall defining a relatively large air space, the inner surface of the wall being generally frusto-conical and disposed with its axis vertical and its small end down, a cylindrical end section at the upper end of the wall having an outlet port therein, an outlet conduit leading from the port, means for introducing pulverulent material into the container near the lower end of the wall, a squirrel-cage beater mounted for rotation on the axis of the wall and having blades extending longitudinally of the wall and end section from a point near where the material is introduced to a point above the bottom of the outlet.

Centrifugal abrading machine. D. C. Turnbull (American Foundry Equipment Co.). U. S. 2,204,635, June 18, 1940 (Feb. 5, 1934; Aug. 4, 1938).

Centrifugal abrasive blasting machine. J. E. Guendling (American Foundry Equipment Co.). U. S. 2,204,587, June 18, 1940 (June 25, 1938).

Conveyer. A. C. Sherrill (Pittsburgh Plate Glass Co.). U. S. 2,204,828, June 18, 1940 (May 6, 1938).—A car-loading device comprises a pair of helically curved conveyer arms each mounted to be swung in substantially circular arcs through the door of a car but in opposite directions, the arcs intersecting, and an auxiliary conveyer for supplying matter to be loaded to the conveyer arms adjacent to intersection of the arcs.

Direct impulse sandblast system. L. D. Peik (American Foundry Equipment Co.). U. S. 2,204,618, June 18, 1940 (Oct. 12, 1932; renewed Aug. 12, 1939).

Drying apparatus for roofing tile and other thin-walled clay plates. Erduin Schondorff. Ger. 683,893, Oct. 26, 1939 (Feb. 28, 1935); V/82*a*. 26. Addition to Ger. 614,369, June 6, 1935. D.A.B.

Electric melting furnace. C. F. Weisgerber (Alloy Steel and Metals Co.). U. S. 2,202,362, May 28, 1940 (Jan. 4, 1938).

Electrical rotating tube furnace for fusing quartz and other hard-melting materials. Soc. Anon. des Manufactures des Glaces & Produits Chimiques de St. Gobain, Chauny & Cirey. Ger. 681,809, Sept. 7, 1939 (March 18, 1933); VI/32*a*. 35.—Quartz and similar high-melting materials are melted in an electric rotating tube furnace by means of a rod-shaped resistance fixed with a spring device; this resistance lies or runs parallel to the axis of the furnace. The cross section of the resistance rod is diminished at both ends, and the rod is held at one end in a current contact plate or post by a rod and a device acting as a key regulable from the outside. The rod, together with the current contact plate or post, can be inserted into the furnace and withdrawn without altering the position of the furnace. D.A.B.

Extruding machine for clay, soap, and other plastic materials. A. F. Wills (W. & F. Wills, Ltd.). U. S. 2,205,328, June 18, 1940 (Feb. 14, 1939).

Heating element mounting construction. E. L. Smalley (Hevi Duty Electric Co.). U. S. 2,202,874, June 4, 1940 (Aug. 3, 1937).

Machine for cutting brick, etc. R. H. Stahl. U. S. 2,205,313, June 18, 1940 (June 5, 1939).

Material-feeding apparatus and method. W. H. Venable (Blaw-Knox Co.). U. S. 2,205,324, June 18, 1940 (June 30, 1937).—Mechanism for apportioning materials into a batch.

Pottery-loading device. A. L. Schreiber (Vernon Potteries, Ltd.). U. S. 2,205,178, June 18, 1940 (Jan. 23, 1939).—A system for handling ceramic ware during manufacture.

Rotary kiln. L. J. Mueller (Standard Lime and Stone Co.). U. S. 2,204,145, June 11, 1940 (Sept. 30, 1938).

Vermiculite-processing machine. W. H. D. Hinchman (Zonolite Co.). U. S. 2,203,821, June 11, 1940 (March 2, 1938).—A machine for separating fragments of exfoliated vermiculite from debris such as stones and unexfoliated fragments of vermiculite comprises an inclined runway along which the exfoliated vermiculite and debris move by gravitation and a closed circuit for effecting a flow of air across the runway.

Volumetric feeder. J. M. Montgomery (Permutit Co.). U. S. 2,204,097, June 11, 1940 (July 15, 1937).—Apparatus for measuring and dispensing fluent materials volumetrically.

Kilns, Furnaces, Fuels, and Combustion

Combustion of hydrocarbons. D. T. A. TOWNEND. *Jour. Inst. Chem. Gt. Brit. & Ireland*, **1940**, Part II, p. 120.—T. reviews work carried out during the present century on the slow and explosive combustion of hydrocarbons. Reference is made to recent work on cool flame formation and to explanatory theories of intermediate peroxides. K.W.W.B.

Discussion on "Stability of a kiln stack." F. H. NORTON. *Jour. Amer. Ceram. Soc.*, **23** [7] 220 (1940); see *ibid.*, [5] 152–56. **Reply.** T. W. GARVE. *Ibid.*, [7] 220.

Formula for calculating the calorific value of coals. Y. KASAKA. *Jour. Fuel Soc. Japan*, **19** [210] 212–22 (1940). —There are many formulas for estimating the calorific value of coal from proximate analysis. Gmelin's, Goutal's, Schmidt's, Whitaker's, Kent's, Geidel's, Rehwinkel's, and Lenoble's are widely used, but for Japanese coal these formulas cannot be applied. The following formulas are used in Japan (Q = calorific value (Cal./kgm.), C = fixed carbon (%), A = ash (%), V = volatile combustible matter (%), W = moisture (%), T = low-temperature tar (%), and G = low-temperature gas (%)); Takei's formula: $Q = 8080 - 80A(1 + 15/100)$; Naito's formula: $Q = 81C + 95T + aG - 12W$, where a is the coefficient affected by $C/(T + G + W)$ and changes from 100 to 160; Nakamura's formula: $Q = 78C + a(V + 0.1A)$, where a is the coefficient for volatile matter and agglutinating power and ranges from 60 to 125; Arai's formula: $Q = f(100 - A - W)$, where f is the variable factor changing with fuel ratio and moisture content. K. reviews and discusses these formulas and suggests a new formula: $Q = 81C + (96 - aW)(V + W)$, where a is the variable factor; when W is less than 5.0, a is 6.5, and when W is equal to or greater than 5.0, a is 5.0. With this formula K. determined the calorific values of 110 coals of Japan and Manchukuo and compared them with those obtained by actual measurements. He states that the calorific value can be estimated with an accuracy of 2% by means of his suggested formula. Y.K.

Graphite electrodes. J. FORSSELL. *Tek. Tid.*, **69** [19] Kemi 33–35 (1939).—F. discusses the physical and electrical properties of graphite and amorphous carbon electrodes for use in electric furnaces working at temperatures above 1200°C. He refers to Arnfelt's investigation of an amorphous carbon electrode, one end of which had been heated sufficiently to convert it completely to graphite while the other end was kept cold, thereby establishing a temperature gradient along the rod. An X-ray examination of this at different distances from the ends showed that graphitization is nothing else than a process of crystallization, the crystals being submicroscopic in size. Further research has proved that the densest arrangement of carbon atoms occurs in the carbonization of organic matter such as pitch, and carbon produced in this way is therefore the most suitable material for making electrodes. F.'s comparison of the two kinds of electrodes shows that those made of graphite offer distinct advantages in electric-arc furnaces, while the cheaper carbon electrode is more economical in reduction furnaces in which the electrodes are actually in contact with the charge. R.A.R.

Hydrogenation of high-volatile bituminous coals. Proximate analysis and characterization of products. L. L. HIRST, H. H. STORCH, C. H. FISHER, AND G. C. SPRUNK. *Ind. Eng. Chem.*, **32** [6] 864–71 (1940).—Results of coal hydrogenation assay work in the liquid phase are presented for three bituminous coals of high-volatile A, B, and C rank. These coals are from the Mary Lee bed of Alabama, the No. 6 bed of Illinois, and the McKay bed of Washington. Analytical data are given on the products of hydrogenation of these coals and of Pittsburgh seam coal. Organic raw materials of industrial importance may be produced from coal by hydrogenation. Illustrated. F.G.H.

Plastic properties of bituminous coking coals. Effect of petrographic composition. R. E. BREWER, C. R. HOLMES, AND J. D. DAVIS. *Ind. Eng. Chem.*, **32** [6] 792–97 (1940). —Relations between the petrographic composition and the plastic properties of six representative bituminous coking coals and of thirteen characteristic layer samples of these coals are studied. The six composite coals (one predominantly splint and five predominantly bright) contained 25 per cent area or more of anthraxylon and had an anthraxylon to attritus ratio of 0.35 to 1 or more. The same is true also of one predominantly semisplint and six predominantly bright layers. The six 100% splint layer samples had an anthraxylon content and anthraxylon to attritus ratio lower than these values. All samples, except three of these 100% splint layers, showed well-defined fusion characteristics by the Davis plastometer test. These three also were lowest in anthraxylon content and anthraxylon to attritus ratio. In general, the fusion temperatures increased with the rank of the coal, but the relative order within a given coal series varied with the predominating type of coal in the given sample. The relative order of decrease in expansion by the Agde-Damm dilatometer test for a typical low-volatile coal series was from bright coal layer to the mixed sample of the entire column to splint coal layer. Except for the predominantly semisplint sample, the coals in each of the six coal series increased in dry, mineral-matter-free total carbon with decrease in bright or increase in splint-coal content. Illustrated. See "Measurement . . .," *Ceram. Abs.*, **18** [8] 220 (1939). F.G.H.

***P-V-T* relations for propane.** WALTER W. DESCHNER AND GEORGE GRANGER BROWN. *Ind. Eng. Chem.*, **32** [6] 836–40 (1940).—Illustrated. F.G.H.

Recent investigations of the Northern Coke Research Laboratory. RILEY. *Jour. Inst. Chem. Gt. Brit. & Ireland*, **1940**, Part II, p. 129.—A lecture dealing with the work carried out at the Laboratory on the carbonization of various materials is summarized. By a study of their diffraction patterns, variations of spacing within and between the layer planes with crystallite size have been detected. The effect of carbonization temperature on these differences in size of crystallites has been examined up to about 2000° with graphite, cokes, coals, and cellulose, and certain generalizations are arrived at. The work is being extended to higher temperatures. The allied question of coalification is mentioned, and certain published theories are qualified. K.W.W.B.

Theory of gas producers based on the laws of equilibria. HECTOR PAQUAY. *Chaleur & Ind.*, **20** [227] 263–70; [228] 316–22, 332–34; [229] 363–64; [231] 461–66 (1939). —After discussing the theories of van't Hoff, Boudouard, and Le Châtelier regarding the reaction carbonic acid plus carbon giving carbon monoxide, P. concludes that there is no equilibrium in gas producers due to the short reaction, imperfect contact between gas and solid fuel, and varying temperature in the gas producer at the bottom and the top. For best results, the thickness of the coke layers, the size of the coke, and the flow of gas must be coordinated. The report is followed by a discussion and the author's reply. F.E.S.

SEPARATE PUBLICATIONS

Annual Report of Research and Technologic Work on Coal, Fiscal Year 1939. A. C. FIELDNER AND R. E. BREWER. *U. S. Bur. Mines Information Circ.*, No. 7105, 59 pp. Free.—A synopsis of the papers published and other work in progress in the Mining, Nonmetals, and Explosives Divisions of the Technologic Branch during the fiscal year ended June 30, 1939, is presented. R.A.H.

Combustibility of Coke in Air. R. E. ZANE AND H. F. YANCEY. *U. S. Bur. Mines Repts. Investigations*, No. 3509, 8 pp. Free.—A working method for evaluating the reactivity or combustibility of coke is described. The sample is weighed as it burns, the loss in weight and time being indicated by a balance and clock arrangement. R.A.H.

PATENT

Protection for controlled-atmosphere furnaces. E. K. HANSEN (Westinghouse Electric & Mfg. Co.). U. S. 2,205,258, June 18, 1940 (Nov. 15, 1939).

Geology

Base exchange in soils: II, Exchange acidity. A. N. PURI AND H. L. UPPAL. *Soil Sci.*, **49** [2] 155–62 (1940).—The phenomenon of exchange acidity in soils has been studied. The results show that the production of free acid on the addition of a salt to an acid soil is governed by the law of distribution of a base between two acids of unequal strength. For Part I see *Ceram. Abs.*, **18** [5] 135 (1939). G.R.S.

Beneficiation of spodumene rock by froth flotation. JAMES NORMAN AND E. W. GIESEKI. *Amer. Inst. Mining Met. Engrs. Tech. Pub.*, No. 1161; *Mining Tech.*, **4** [2] 9 pp. (1940).—Spodumene is a lithium-bearing pyroxene. It may be desirable in glass batches, and its use in ceramics will increase as mixtures of it with feldspars have a lower maturing temperature than a feldspar alone. It ordinarily occurs with quartz, feldspar, and mica. Rock from near the surface, stained with iron and manganese oxides, did not float. To clean the rock, attrition scrubbing was used with cleaning reagents such as Na_2SiF_6, Na_3PO_4, Na_2S, and NaOH. NaOH alone or with Na_2S was best; almost any anionic collector gave satisfactory concentration of spodumene. Spodumene in rocks from six localities was satisfactorily floated using oleic acid after proper cleaning. A concentrate of 90 to 95% spodumene was prepared, and the tailings had about 1% spodumene. To avoid difficulties with the iron-containing mica, both were floated by using Armour Amine AM-1180, and the spodumene was then depressed by sulfuric acid. Feldspar can be separated from the spodumene tailing by the use of hydrofluoric acid and lauryl amine hydrochloride. A spodumene-feldspar concentrate can be made from the crude rock by the same reagents or, preferably, by the addition of oleic acid first and then the other reagents. W.D.F.

Development and growth of the feldspar industry. H. B. DUBOIS. *Bull. Amer. Ceram. Soc.*, **19** [6] 206–13 (1940).

Electrophoresis, electroendosmosis, viscosity, and ζ-potential of suspensions of Japanese acid clay. KEN-ITI YAMAMOTO. *Waseda Applied Chem. Soc. Bull.*, **13** [29] 18–29 (1936).—Y. studied the capillary electrical phenomena of Japanese acid clay and made the same experiments on fuller's earth, kaolin, silica gel, ferric hydroxide gel, and colloidal carbon as a comparison. The velocity of electrophoresis of Japanese acid clay and other suspensoids was determined by Burton's U-tube method. The migration velocity of electrophoresis of acid clays lies between 39.9×10^{-5} and 43.9×10^{-5} cm./sec. The velocities of electroosmosis of Japanese acid clay are between 0.97 and 1.48×10^{-5} cc./sec. To obtain the ζ-potential of Japanese acid clay, the viscosity was determined with the Ostwald viscosimeter. The ζ-potential of Japanese acid clay lies between -0.066 and 0.058 volt. Y.K.

Investigation of the composition of bauxite rocks. I. N. MASLENITSKII. *Zavodskaya Lab.*, **8**, 933–36 (1939).—The method is based on the principle of selective dissolution of the bauxite components. The most adequate reagents for the separation of hydrargillite are (1) HCl acid (1.19%), (2) N alkali solutions, and (3) H_2SO_4 (70%). The results obtained are not absolutely correct owing to the solubility of the other bauxite components in these reagents. The method should be supplemented by physical methods of investigation. P.B. & E.S.

Laws of soil colloidal behavior: XXIA, Amphoteric points, the pH, and the Donnan equilibrium. Theoretical. SANTE MATTSON AND LAMBERT WIKLANDER. *Soil Sci.*, **49** [2] 109–53 (1940).—The property of amphoteric soils to exchange H and OH ions simultaneously for the cations and anions of a neutral salt solution at or near the equiionic point of the soil and the valence effect in the mass law (as expressed by the Donnan distribution of ions between the soil complex and the soil solution) lead to some very significant, but heretofore neglected, consequences. For the salts Na_2SO_4 and $CaCl_2$, the following expressions apply: for acidoids, $\frac{(H^+)_i}{(H^+)_o} = \frac{(Na^+)_i}{(Na^+)_o} = \frac{\sqrt{(Ca^{++})_i}}{\sqrt{(Ca^{++})_o}}$, and for basoids, $\frac{(OH^-)_i}{(OH^-)_o} = \frac{(Cl^-)_i}{(Cl^-)_o} = \frac{\sqrt{(SO_4^{--})_i}}{\sqrt{(SO_4^{--})_o}}$, where the parenthesis represents activity and i and o signify that the ions are in the inside and the outside solution, respectively. The fact that the divalent ions enter the equation in the form of the square root of their activity indicates that, in low concentrations (in the outside solution) of the salt and in the presence of equivalent proportions of free acidoids and basoids, the number of H ions displaced by the Ca ions will be greater than the number of OH ions displaced by the Cl ions and the number of OH ions displaced by the SO_4 ions will be greater than the number of H ions displaced by the Na ions. In high concentrations, the displacing power of the different ions will approach the same value. These conclusions are all drawn with the proviso that none of the ions enter into any specific reaction. From this it follows that, under these conditions, $CaCl_2$ (or a salt of the type $M^{++}S_2^-$) must yield a maximum in exchange acidity in dilute solutions and Na_2SO_4 (or a salt of the type $M_2^+S^{--}$) must yield a maximum in exchange alkalinity in dilute solutions. A theoretical study has been made of the various factors which determine the position and magnitude of these maxima, such as the acidoid/basoid ratio, the concentration of colloid, and the concentration of the ions in the micellar solution. By means of titrations and pH determinations of different soils in various concentrations of salts, it is possible to prove the application of the theory. See *Ceram. Abs.*, **19** [4] 99 (1940); for Part **XX** see *ibid.*, **17** [1] 43 (1938). G.R.S.

Montmorillonite in fuller's earth, Nutfield, Surrey. A. BRAMMALL, J. G. C. LEECH, AND W. J. DUNSTALL. *Geol. Mag.*, **77**, 102–12 (1940).—A sample was deflocculated in water, and the upper part was siphoned off, washed, and dried. This was 76 to 90% montmorillonite; other minerals are quartz, amorphous silica, free alumina, free ferric oxide, sphene, variable feldspars, and barite. The chemical analysis was corrected for these and compared with standard montmorillonite analyses. The base-exchange capacity was measured by acid treatment. Dehydration curves are given. W.D.F.

Ohio's dolomites and limestones. WILBER STOUT. *Ohio State Univ. Eng. Expt. Sta. News*, **11** [2] 8–10 (1939).—These rocks appear near the surface under 17,000 sq. miles and have a total thickness of 2400 ft. They have over 200 definite uses. They are used in construction, in smelting iron ore, as a flux, in refractories, glass, and Portland cement, as lime, in hundreds of chemical industries, in agriculture, and in medicine. These formations range from the Ordovician to the Devonian. W.D.F.

Philippine chromite. DEAN F. FRASCHE. *Mining Congr. Jour.*, **25** [12] 22–27 (1939).—Large areas of widely scattered chrome-bearing ultrabasic rocks are principally confined to two belts along the eastern and western borders of the islands. Only an insignificant part of the chrome-bearing areas has been systematically prospected. Zambales province in western Luzon has the most extensive proven reserves. At the Consolidated Mines, Inc., development shows 10,000,000 metric tons of ore, the largest known reserve. The ore mineral is a high-alumina chrome spinel containing about 33% Cr_2O_3, and it is sold as a refractory. It is trucked 27 kilometers to the port of Masinloc. The Acoje Mining Co. has the second largest reserves, 67,000 tons of metallurgical ore (more than 46% Cr_2O_3) and 88,000 tons of lower grade. Other occurrences of chromite not in this district are on Dinagat Island north of Mindanao, northern Mindanao, the southern end of Samar Island, and Homonhon Island. The most recently discovered deposits are on Ambil Island, northwest of Mindoro. Most of the ore is mined by open-cut methods.

Production in 1938 was 67,000 metric tons, of which 54,000 tons were shipped to the U. S. W.D.F.

Phosphate deposits of the United States. GEORGE R. MANSFIELD. *Econ. Geol.*, **35** [3] 405–29 (1940).—Different types of phosphate deposits and sources of phosphate in the U. S. are briefly discussed. Reserves of the U. S. as a whole are now considered to exceed ten billion tons, not counting those classed as possible phosphatic limestones. At the present rate of consumption (3,000,000 tons annually), 10 billion tons would last more than 3000 years. J.L.G.

Physical characteristics of soils: VI, Influence of clay, exchangeable bases, and hygroscopic moisture on soil cohesion. A. N. PURI, A. G. ASGHAR, AND A. N. DUA. *Soil Sci.*, **49** [3] 239–49 (1940).—Cohesion for the same percentage of clay falls rapidly as the particle size increases up to a limit beyond which the effect of the size of particles becomes negligible. In single-base soils, the order of cohesion for the dry soil follows the generally accepted order of dissociation for these ions. For Part II see *Ceram. Abs.*, **18** [5] 136 (1939). G.R.S.

Primary minerals of the silt fraction as contributors to the exchangeable-base level of acid soils. E. R. GRAHAM. *Soil Sci.*, **49** [4] 277–81 (1940).—A study of the influence of hydrogen clay on the metathesis of the bases from the structure of primary minerals of the silt size revealed that, in the comparatively short interval of 107 days, calcium can be transposed from the crystal of anorthite to the exchange atmosphere of hydrogen colloidal clay in such quantities as to change the pH of soil clay from 3.30 to 5.70. The hydrogen of the colloid can be effective in bringing about calcium removal also from the crystals of hornblende and augite. Biotite and microcline are only slightly affected by the action of the hydrogen sorbed in the exchange atmosphere of colloid clay. The action of sorbed hydrogen ions of the clay brought about a weathering effect on the mineral crystal significant enough to remove 3.4% of the total calcium held in the anorthite mineral. The hydrogen clay thus becomes an active agent in the weathering processes when it causes approximately 100 times as much calcium to be removed from anorthite as was removed by the hydrolytic action of water. G.R.S.

Purification of diatomite by froth flotation. JAMES NORMAN AND OLIVER C. RALSTON. *Amer. Inst. Mining Met. Engrs. Tech. Pub.*, No. 1198; *Mining Tech.*, **4** [3] 11 pp. (1940).—The authors attempted to develop a method by which eastern users could obtain diatomite from low-grade eastern deposits. The material was from the Patuxent River deposits at Dunkirk, Md., and was roughly one-third each of clay, quartz, and diatomite. For dispersing the clay, heating with live steam at 90°C. for 30 min. and cooling for 30 min., with an addition of sodium carbonate and sodium hydroxide, gave the best results. The clay was floated best by using diamylamine diluted with pyridine and crude oil as collector. Lompoc, Calif., diatomite was amenable to the same treatment. Other diatomites from arid localities were more difficult to treat, as the clay had been baked onto their surfaces. These had to be treated in the attrition mill three times and allowed to settle 24 hr. after each treatment; then heating with hydrofluoric acid to disperse clay and sodium hypochlorite and sulfuric acid to oxidize organic matter was most effective. Lauryl amine hydrochloride had to be used as collector. Goodland, Kansas, calcite diatomite was dispersed with sodium lignin sulfonate and floated with oleic acid and pine oil. W.D.F.

Surface tension and thixotropic properties of Putnam clay suspensions. J. W. BELTON. *Proc. Leeds Phil. Lit. Soc., Sci. Sect.*, **3**, 565 (1939).—The surface tensions of electrodialyzed Putnam clay were measured by the bubble-pressure method, using scrupulously clean apparatus. In a sample of clay (I) containing varying sized particles, the pressure required to blow a bubble increases almost linearly with time. Another sample (II) of electrodialyzed clay divided into five fractions according to particle size (50 mμ, 100 mμ, 100 to 200 mμ, 200 to 500 mμ, and 1μ to 500 mμ) showed this phenomenon in only one particle size fraction, *viz.*, 100 to 200 mμ. The surface tension of colloidal solutions is known to vary with time; thus, a gelatin solution showed this effect. Blowing bubbles through the solution brought it back to its original value, suggesting that a structure is built up. In one case with clay I, the removal of the jet from the suspension appeared to inhibit the increase with time, but the opposite was the case with the 100 to 200 mμ fraction of clay II. It appears that clays in the absence of electrolytes may be thixotropic, contrary to the opinions of previous workers. L.R.B.

Turkish chrome ores. ANON. *Der Südosten; Tek. Tid.*, **69** [14] Bergvetenskap 30–31 (1939).—Brief particulars are given of the occurrence and production of chrome ores in Turkey. The quantity of ore exported has risen from a negligible quantity in 1928 to 150,000 tons in 1936. The largest occurrence is at Ergani in an area 30 x 18 kilometers. The ore contains about 48 to 58% of chromic oxide. R.A.R.

Zettlitz kaolin. ANON. *Ceramica*, **2** [3] 109–13 (1940).—No substitute has yet been found which possesses the superior chemical, physical, and ceramic characteristics of Zettlitz kaolin for the manufacture of quality products. The chemical, physical, and ceramic properties of Zettlitz kaolin, Edel-Osmose kaolin, "imperial" kaolin, Meka kaolin, Oka kaolin, and Börtewitz kaolin are listed. Kaolin is also used in other industries where its reactivity and behavior when introduced in powdered form, its degree of dispersion and grain size, specific weight, hardness, chemical composition, and particle form are of prime importance. In the manufacture of paper, kaolin (especially kaolin "T" and "OP") was found useful as a filler of the proper grain size and as an aid to cohesion. Kaolin is also used as a filler in the manufacture of colors ("imperial" kaolin for blue ultramarine and Zettlitz, Edel-Osmose, and "imperial" kaolins for colored crayons or pencils according to the quality desired) and in textiles, linoleum, soaps, and chemicopharmaceutical preparations. Colloidal kaolin, which is well suited to these special uses, has been specially prepared; it contains 90% colloidal particles with a diameter of less than 1 micron. This kaolin is made especially for the rubber industry where it is better distributed when added to the rubber and imparts a greater durability and resistance; it absorbs the same color as the rubber. M.V.C.

BOOKS AND SEPARATE PUBLICATIONS

Adirondack Igneous Rocks and Their Metamorphism. A. F. BUDDINGTON. Geological Society of America, New York. 354 pp. Price $3.25. Reviewed in *Science News Letter*, **37** [13] 208 (1940).—B. describes in full detail the geology of one of the most interesting and the older geologic areas of this country. P.G.H.

Domestic China Clay Sales Make New Record in 1939. ANON. *U. S. Bur. Mines Mineral-Market Rept.*, MMS 813, 2 pp. Free. R.A.H.

Geology and Engineering. ROBERT F. LEGGET. Foreword by P. G. H. Boswell. McGraw-Hill Book Co., Inc., New York, 1939. 642 pp., 225 figs. Price $4.50. Reviewed in *Econ. Geol.*, **35** [3] 471–72 (1940).—This book is recommended for advanced students or practicing engineers who already possess the fundamentals of geology. J.L.G.

Outline of Mineralogy. LOUIS F. LEONARD. Edwards. 88 pp. Price $2.50. Reviewed in *Science News Letter*, **37** [10] 160 (1940).—L. gives the properties and a general description of minerals. Supplementary tables of hardness, color, etc., aid in the identification of specimens. P.G.H.

Properties and Numerical Relationships of the Common Elements and Compounds. J. E. BELCHER AND J. C. COLBERT. D. Appleton-Century Co., Inc., New York. 350 pp. Price $2. Listed in *Science News Letter*, **37** [17] 272 (1940). P.G.H.

PATENTS

Decolorizing of clays. ENGLISH CLAYS, LOVERING, POCHIN, & CO., LTD., AND R. J. DAVIES. Brit. 520,498, May 8, 1940 (Oct. 24, 1938).

Manufacture of synthetic mineral products. W. Ditterichs. Brit. 520,889, May 22, 1940 (Oct. 30, 1937).

Opening up of zirconium ores. I. G. Farbenind. A.-G. Brit. 520,740, May 15, 1940 (Oct. 30, 1937).

Chemistry and Physics

Applications of X-ray technique to industrial problems. H. P. Rooksby. *Jour. Roy. Soc. Arts*, **88** [4552] 308–38 (1940).—Of the several methods in use for determining crystal structure by means of X-rays, only one is widely used in industry for analytical purposes. This is the powdered crystal method, discovered independently by Hull in America and by Debye and Scherrer in Germany. In general, each crystalline chemical compound has a unique structure or arrangement of atoms in space and will accordingly have a characteristic X-ray diffraction pattern. R. explains how such a pattern is secured by the use of cadmium oxide. The X-ray apparatus recording the pattern is shown together with the powdered crystal photograph derived, which somewhat resembles that of a spectrum on a spectrographic plate. An unknown chemical compound may also be identified. By chemical or spectroscopic analysis, the elements present may be ascertained; X-ray diffraction methods will then reveal how these elements are combined. In the study of refractory materials, the X-ray technique is useful in following the changes in the state of combination of the constituents; the heat treatment to which a mixture of aluminum and silica must be subjected to obtain complete formation of the aluminum silicate mullite ($3Al_2O_3 \cdot 2SiO_2$) may be determined. No chemical method can give this information. Several American workers have attempted to classify data on X-ray diffraction patterns in terms of interplanar spacings, hoping to arrive at a rapid method of identification somewhat like that of identifying the lines on a spectroscopic plate from tables of wave length. Keys of powder diffraction data for, 1000 chemical substances and an index for rapid reference were made. The X-ray method is also useful when only a small quantity of material is available for examination. R. points out the use of the method in opal glasses where crystalline inclusions occur or where glass articles for domestic purposes are impaired by crystalline matter (stones). Knowledge of the origin of these defects and their particle composition, as determined by X rays, leads to intelligent processes of removal. The X-ray method of examination may be applied to changes in the crystalline forms of silica. Alumina also has different crystalline modifications, two of the trihydrate, two of the monohydrate, and two of the anhydrous form, all shown by their X-ray patterns. Different fluorescent colors are sometimes associated with change in the crystalline form. The usual fluorescent color of zinc orthosilicate is green, but under different conditions of preparation it may be yellowish; X-ray patterns have demonstrated that the colors are associated with two different crystal forms. The X-ray pattern has been used as an identification mark in filing a patent claim for the second variety of fluorescent zinc silicate, the first use of X-ray diffraction measurements for identification purposes in patent literature. A discussion is included. Ten illustrations. K.R.

Contact angles of "built-up" multilayers. J. J. Bikerman. *Trans. Faraday Soc.*, **36** [2] 412–17 (1940).—A new method of determining large wetting angles is based on the measurement of the marks left by drops on solid surfaces. Wetting angles between air, water, and "built-up" multilayers were measured. They confirm the belief that *Y* films are deposited when the angle is near 90°, more hydrophobic surfaces give rise to *X*, and less hydrophobic angles give *Z* deposition. Langmuir's hypothesis connecting wetting angles with molecular orientation and the usual theory of contact angles are briefly discussed. G.R.S.

Crystal chemistry of nonmetallic inorganic substances: II. F. Machatschki. *Naturwissenschaften*, **27** [40] 670–74; [41] 685–93 (1939).—M. gives a review of recent developments in the knowledge of crystal structure, dealing with elements, compounds of crystal chemically heterogeneous components, e.g., the cubic high crystobalite ∞ (SiO_2) and others, and, among the group of complicated compounds, complex silicates, glasses, aluminates, tungstates, etc. Bibliography. J.M.N.

Detection of lime in magnesite. F. Schwarz. *Mikrochim. Acta*, **3**, 126–28 (1938); abstracted in *Referat. Silikatliteratur*, **6** [3] 6239 (1939).—F. Feigl and H. Leitmeier worked out a simple method for detecting magnesium carbonate in rock with diphenylcarbazide; they also described briefly a color reaction with alizarin solution for the detection of calcite and dolomite in magnesite. According to these two methods, a cut surface or a granulated sample is immersed in a heated alkaline solution of diphenylcarbazide. Magnesite turns red and calcite and dolomite remain white. If the specimens are immersed in a boiling solution of alizarin sulfoacid sodium (0.1%) and hydrochloric acid ($\sim 1/_{15}$ *N*), the lime forms a violet lake, while magnesite is colored first yellow and later pale violet. This determination is about ±1% exact. Both reactions occur on crude rock. R. Rieke and O. Wiese (*Ceram. Abs.*, **7** [9] 625 (1928)) found lime and magnesite by coloring the calcined samples with *p*-nitrobenzene-azoresorcinol; the lime became yellow-brown and the magnesia dark olive green. A differentiation between calcite and dolomite in caustic fired magnesite is made by dabbing or spraying the samples with phenolphthalein or thymolphthalein. The determination of magnesite is feasible when calcite and dolomite feldspar are inactive. In caustic magnesite part of the calcium is present as active CaO. A rapid method which permits the calcium present (active lime) to be determined separately as calcium oxide is given: caustic magnesite which contains active lime is moistened with a few drops of $1/_2$ *N* ammonium citrate solution on a watch glass or slide and is then dabbed with thymolphthalein. After a few seconds the CaO grains become blue. Grains of 0.05 mm. density can be observed with a microscope. Calcite and dolomite do not react under these conditions. In the determination of the caustic ingredients (CaO and MgO) in dead-burned sinter magnesite where the lime is contained as oxide, silicate, or ferrite, the lime reacts as CaO with ammonium citrate and thymolphthalein as in caustic burned magnesia. The amount of MgO and CaO is visible when sprayed with phenolphthalein. The incompletely sintered MgO grains show red coloring in the form of minute efflorescences under a microscope. Since sinter acquires more or less activity according to the length of calcining time or recovers it when stored, the length of storing time of sinter magnesite and the suitability of further working can also be studied by these reactions. M.V.C.

Determination of boron in small amounts and traces. O. Unverdorben and R. Fischer. *Bodenkunde & Pflanzenernähr.*, **13**, 177–94 (1939).—Gravimetric, volumetric, colorimetric, spectroscopic, and polarizing methods are briefly discussed. The authors base their experiments upon the volumetric research method of Schulek and Vastagh, as the best for estimating the smaller and smallest quantities of boron. They discuss the preparation of a soda lye absolutely free from carbonic acid, as only by the use of such a product can correct values be guaranteed. Differences between their procedure and that hitherto recognized are given. Special titrating flasks of boron-free glass with soda-lye tubes and titration under soda-lye closure prevented any possible entrance of CO_2 from the air and, by requiring only one indicator, eliminated much of the error. By these changes only published values of boron traces would be questioned. They preferred to titrate phenolphthalein against naphtholphthalein since their experience in changing white to rose would enable the traces to be more easily recognized than by the long conversion of yellow over green to blue. The authors proved in their estimation that smaller and smallest boron

quantities must unconditionally increase the gross weight in raising the average homogeneous standard. The methyl alcohol-sulfuric acid distillation was also found to increase the gross weight. Numbers for weighing-in are named which would be eventually correct for weights in boron estimation if the results corresponded to factual conditions and were not merely probable. The following results were obtained: (1) separation of boron as boric acid methyl ester through distillation; (2) decomposition of sulfite, formate, and acetate; (3) exclusion of CO_2; and (4) titration of the isolated boric acid with only one indicator and under soda-lye closure. Pertinent researches were studied to determine all eventual influences which might break down the titration estimates. This paper merely gives the procedure of the authors; their numerical results will be published later. 23 references. K.R.

Determination of minute amounts of potassium. Iodometric evaluation of the cobaltinitrite precipitate, using ceric sulfate. Irving Allen Kaye. *Ind. Eng. Chem., Anal. Ed.*, **12** [5] 310–11 (1940). F.G.H.

Determination of the total sulfur in coal and coke by reduction with a subsequent rapid analytical method. W. Mantel and W. Schreiber. *Glückauf*, **75** [48] 929–36 (1939).—To determine the S content, often undesirable in combustion gases from coal and coke (especially for protective atmospheres), as correctly and rapidly as possible, a new method was developed by which S is not oxidized but is reduced with calcium oxalate, CaOH, Li_2CO_3, and Cd acetate in the gasified coal and coke. The exact procedure for coke and coals with less than 30% and over 30% volatile constituents is given in detail, and the apparatus used is described. A determination takes 20 to 30 min. and gives an accuracy of ±0.03%. M.H.

Extraction of alumina from aluminum phosphate ore: I. Kei-iti Akiyama. *Waseda Applied Chem. Soc. Bull.*, **16** [38] 11–17 (1939).—A. extracted alumina from aluminum phosphate ore by the following process: The aluminate slag which is obtained by fusing aluminum phosphate ore with lime and coke in the electric-arc furnace is digested with a sodium carbonate solution containing a small quantity of sodium hydroxide; sodium aluminate solution is thus obtained. Carbon dioxide is added to this solution, and, after long agitation, the pure aluminum hydroxide is precipitated. When the alumina to silica ratio of the ore is less than 4, the chemical composition of the aluminate slag is similar to that of the lime-alumina cement. From such slag, only about 30 to 45% alumina can be extracted. A. suggests increasing the alumina-silica ratio of the ore to 4 by adding diaspore or bauxite. Y.K.

Extraction of vanadium salts: I, Separation of vanadium from ferrous iron. S. Uno and T. Izumo. *Waseda Applied Chem. Soc. Bull.*, **13** [3] 1–6 (1936).—The authors determined the hydrogen-ion concentration when the aqueous solution of alkali is added to the acidic solution of vanadium sulfate by an antimony electrode (Sb 0.1 *N* calomel cell against H_2 0.1 *N* calomel cell). The precipitation of $H_4V_2O_8$ occurs when the pH value lies between 4 and 5. The same experiments were carried out for ferrous sulfate solution; the results were similar to those obtained by H. T. S. Britton, and the precipitation of iron hydroxide occurs when the pH lies between 6 and 8. When the mixed solution of vanadyl sulfate and ferrous sulfate is neutralized with alkali to pH 5 to 6, the hypovanadic acid can also be completely separated from the large amount of ferrous salt. The hypovanadic acid can not be precipitated completely from a dilute solution; this fact can be accounted for by the colloidal properties of hypovanadic acid. It is therefore necessary to study the coagulation of hypovanadic acid. Y.K.

Fusibility diagram of the system ZrO_2–MgO. N. A. Zhirnova. *Zhur. Priklad. Khim.*, **12**, 1278–86 (1939).—The liquidus line in the system ZrO_2–MgO has been determined. The existence of one eutectic point corresponding to 49 mol. % MgO at 2070° and the absence of the maximum indicated by Wartenberg and Werth have been confirmed. The fusibility diagram of the system ZrO_2–MgO indicates that above the transformation temperature of ZrO_2 (1000°) there is, on the ZrO_2 side, a solution of continuancy with the formation of two regions of solid solutions: the first (β), very limited, possesses a monoclinic form, and the second (α), more expanded, has a cubic form. The practical value of the findings for the production of highly refractory masses is stressed. P.B. & E.S.

Heats of formation of calcium aluminates. W. A. Roth and Ursula Wolf. *Z. Elektrochem.*, **46** [3] 232–33 (1940).—Recent experiments resulted in the determination of the following heats of formation: $5CaO \cdot 3Al_2O_3$ = −11.1 Cal. at 20°C.; $3CaO \cdot Al_2O_3$ = −3.9 Cal. at 20°C. The possible errors are ±1 to 1.5 Cal. J.M.N.

Influence of interfacial curvature on solubility. R. S. Bradley. *Trans. Faraday Soc.*, **36** [2] 392–94 (1940).—If curvature occurs at the interface between two liquids, the solubility is altered. The behavior is considered in the light of a change in energy due to molecular interaction without reference explicitly to the surface pressure due to curvature. The change in energy is caused by the difference in molecular environment between a molecule at a plane and at a curved surface, and the method is advantageous in that the inquiry can be pursued down to small droplets. The results apply not only to the solubility of liquids in liquids but also to the condensation of vapors on nucleating particles or in capillary systems. G.R.S.

Influence of silica, titania, and alumina on the viscosity of acid synthetic slags and the calculation of the viscosity of acid and basic blast-furnace slags from their chemical composition. Kurd Endell and Gunter Brinkmann. *Stahl & Eisen*, **59**, 1319–21 (1939).—The relationship between temperature and viscosity was thoroughly investigated in a previous research by K. Endell and R. Kley (*ibid.*, pp. 677–85). To complete this work, it was necessary to investigate the effect of increased percentages of the major constituents of the slag on the viscosity for any particular temperature. A synthetic slag of the following composition was therefore made up: SiO_2 46.1, CaO 30.5, Al_2O_3 18.4, and MgO 5.1%. The ratio of $CaO:SiO_2$ in this starting slag equals 0.66. For the first experiment the viscosity of the initial slag was measured over the temperature range 1300° to 1500°C. Enough silica was then added to decrease the $CaO:SiO_2$ ratio successively to 0.60, 0.55, 0.52, and 0.48. The viscosity was measured in the same temperature range as the starting slag. The lower the ratio of $CaO:SiO_2$, the higher is the viscosity at any given temperature. When the $CaO:SiO_2$ ratio was 0.55, the viscosity of the slag at 1400°C. was 75 poises. The total percentage of SiO_2 at this point was 50.4%. A similar procedure was followed, except that the percentages of alumina were varied as follows: 18.4% in the starting slag and 25.8, 28, 33.9, and 40.4% in the succeeding slags. The higher the percentage of alumina, the higher is the viscosity of the slag at any particular temperature. For titania, using the same method, it was found that the higher the titania content, the lower is the viscosity. The highest percentage of titania in the slags investigated, however, was 17.3%. The higher the alumina content, the higher was the temperature at which crystallization occurred. A hyperbolic relationship was also discovered which permits the determination of the viscosity of a slag at 1400°C. if the percentages of the constituents are known; a so-called "viscosity number," K_s, is plotted against the viscosity isothermally. K_s =

$$\frac{CaO + MgO + TiO_2 + FeO + MnO + alkalis + sulfides}{SiO_2 + Al_2O_3}$$

All quantities are expressed in weight per cent. K_s varies from 0.5 to 1.4 in the investigation. It covers the range from basic to strongly acid slags. It is thus possible to calculate the viscosity of any slag at 1400°C. if the chemical composition of the slag is known. 5 graphs and 1 table. J.P.S.

Lime–vanadium pentoxide system. A. N. Morozov. *Metallurg*, **13** [12] 21–28 (1938).—An investigation of the

lime–vanadium pentoxide system (CaO–V_2O_5) up to a range of 50% CaO indicates the existence of the compounds $CaO \cdot V_2O_5$, $2CaO \cdot V_2O_5$, and $3CaO \cdot V_2O_5$ having melting points of 778°, 1015°, and 1380°, respectively. B.Z.K.

Measuring the viscosity of the system B_2O_3–SiO_2 and calculation of the activation energy of glasslike systems. A. LEONT'EVA. *Zhur. Fiz. Khim.*, **13**, 1020–23 (1939).—Fusions of B_2O_3 and SiO_2 mixtures evidence several peculiarities; at 800° their viscosity is higher than that of B_2O_3 at the same temperature; with 15% SiO_2, a rise of the 800° isotherm begins. At about 5% SiO_2 all isotherms have a minimum, especially abrupt in the case of the 530° isotherm. A second less sharp minimum exists at 12% SiO_2. The decrease of viscosity is accompanied by an increase of volume. This is not as large as necessary to level the minima of the 530° and 630° isotherms. Viscosity measurements and activation energy calculations showed that the process of dissolution of SiO_2 in B_2O_3 at small concentrations is accompanied by a great dissociation of B_2O_3 and SiO_2 molecules and that in a definite temperature interval (under 800°) a change takes place in the character of the interaction between particles. At concentrations over 10% SiO_2, borosilicate glasses behave as ordinary structural liquids in which, with decreasing temperature, only the molecule coordination changes. P.B. & E.S.

Phase composition of cement clinker in the system $3CaO \cdot SiO_2$–$2CaO \cdot SiO_2$–$3CaO \cdot Al_2O_3$–$4CaO \cdot Al_2O_3 \cdot Fe_2O_3$ at clinkering temperatures: I–IV. L. A. DAHL. *Rock Products*, **41** [9] 48–50; [10] 46–48; [11] 42–44; [12] 44–47 (1938).—D. gives a detailed theoretical discussion of the solution of problems pertaining to the ternary system CaO–Al_2O_3–SiO_2 and the quaternary system. The general principles used are as follows: (1) When a state of equilibrium is attained in which a liquid and one or more solid phases are present, the fractional proportion of liquid is equal to the percentage of any disappearing phase in the total composition divided by the percentage of that phase in the composition of the liquid. (2) When a state of equilibrium is attained in which a liquid and one or more solid phases are present, the fractional proportion of liquid is equal to the sum of the percentages of the disappearing phases in the total composition divided by the sum of the percentages of those phases in the composition of the liquid. (3) When two or more solid phases disappear, the disappearing phases are in the same relative proportions in the total composition and in the compositions of the liquid. (4) As a preliminary step in the computation of phase composition, the compositions of mixture and liquid should be expressed in terms of substances equal in number to the component number of the system, and these should include those substances which are capable of existing as solid phases in equilibrium with the liquid. (5) All mixtures which may be formed from any given series of substances are located within the geometric figure formed by drawing a straight line between the points representing the substances. Equations for the transformation of oxide compositions to the systems $3CaO \cdot SiO_2$–$2CaO \cdot SiO_2$–$3CaO \cdot Al_2O_3$–$4CaO \cdot Fe_2O_3$, CaO–$3CaOSiO_2$–$3CaOAl_2O_3$–$4CaO \cdot Al_2O_3 \cdot Fe_2O_3$, and $3CaOSiO_2$–$3CaO \cdot Al_2O_3$–$5CaO \cdot 3Al_2O_3$–$4CaOAl_2O_3 \cdot Fe_2O_3$ are sufficient for all problems of this type encountered in the study of Portland cement compositions in the ternary and quaternary systems under consideration. These equations are as follows:

SERIES 1: SYSTEM $3CaO \cdot SiO_2$–$2CaOSiO_2$–$3CaOAl_2O_3$–$4CaOAl_2O_3 \cdot Fe_2O_3$

$$3CaOSiO_2 = 4.0710\ CaO - 7.6024\ SiO_2 - 1.4297\ Fe_2O_3 - 6.7187\ Al_2O_3 \quad (1)$$

$$2CaOSiO_2 = 8.6024\ SiO_2 + 1.0785\ Fe_2O_3 + 5.0683\ Al_2O_3 - 3.0710\ CaO \quad (2)$$

$$= 2.8675\ SiO_2 - 0.7544\ 3CaOSiO_2 \quad (3)$$

$$3CaOAl_2O_3 = 2.6504\ Al_2O_3 - 1.6920\ Fe_2O_3 \quad (4)$$

$$4CaOAl_2O_3Fe_2O_3 = 3.0432\ Fe_2O_3 \quad (5)$$

SERIES 2: SYSTEM CaO–$3CaOSiO_2$–$3CaOAl_2O_3$–$4CaO$-$Al_2O_3Fe_2O_3$

$$CaO = CaO - 2.8012\ SiO_2 - 0.3512\ Fe_2O_3 - 1.6504\ Al_2O_3 \quad (6)$$

$$3CaOSiO_2 = 3.8012\ SiO_2 \quad (7)$$

$$3CaOAl_2O_3 = 2.6504\ Al_2O_3 - 1.6920\ Fe_2O_3 \quad (8)$$

$$4CaOAl_2O_3Fe_2O_3 = 3.0432\ Fe_2O_3 \quad (9)$$

SERIES 3: SYSTEM $2CaOSiO_2$–$3CaOAl_2O_3$–$5CaO \cdot 3Al_2O_3$–$4CaOAl_2O_3Fe_2O_3$

$$2CaOSiO_2 = 2.8675\ SiO_2 \quad (10)$$

$$3CaOAl_2O_3 = 3.6133\ CaO - 6.7478\ SiO_2 - 3.3130\ Al_2O_3 - 2.9610\ Fe_2O_3 \quad (11)$$

$$5CaO3Al_2O_3 = 4.8803\ SiO_2 + 4.3130\ Al_2O_3 + 0.9178\ Fe_2O_3 - 2.6133\ CaO \quad (12)$$

$$4CaOAl_2O_3Fe_2O_3 = 3.0432\ Fe_2O_3 \quad (13)$$

The solutions of many problems are described and illustrated. The use of a combination of graphic methods and computations is shown. V–VII. *Ibid.*, **42** [1] 68–70; [2] 46–49; [4] 50–53 (1939).—The behavior of phase compositions when liquids leave quintuple lines as well as after liquids leave quintuple lines is described. The delayed or arrested crystallization, arrested independent crystallization, and partial independent crystallization and the deposition of magnesia, alkalis, and free lime in cement clinker are discussed in detail. Seventy equations are given for calculating phase composition during the process of fusion and the products of complete independent crystallization as well as for the phase composition of clinker with an estimated glass content for normal and partial independent crystallization. Many references, data, and diagrams are given. J.J.H.

Photocolorimetric determination of Fe_2O_3, MnO, and TiO_2 in white Portland cement. E. I. NAGEROVA AND A. D. LEBEDEVA. *Zavodskaya Lab.*, **8**, 1069–73 (1939).—The photocolorimetric method may be used and is to be preferred to the colorimetric method because it is quick and simple. P.B. & E.S.

Photocolorimetric determination of silica in alumina cements. V. T. ILLUMINSKAYA. *Zavodskaya Lab.*, **8**, 863–66 (1939).—The method is based on the attainment of a colored silicomolybdate complex formed by the action of ammonium molybdate on SiO_2 in an acid solution. Picric acid was used for the latter. The M. Z. Kirvov colorimeter was used for the determinations. The method permits the determination of SiO_2 in alumina cements in relatively high concentrations (6 to 10%). The time needed for a determination is 60 to 80 min. P.B. & E.S.

Physics of stressed solids. ROY W. GORANSON. *Jour. Chem. Phys.*, **8** [4] 323–34 (1940).—The internal energy of a system is subdivided into a work or potential function and a thermal or kinetic function (the former expressed in terms of the current electrostatic theory of intercrystalline bonding), and these functions are then examined for variations of temperature, hydrostatic pressure, unidirectional stress, and combined hydrostatic and unidirectional pressure. From these considerations, a theory is evolved which not only seems to explain and correlate satisfactorily the phenomena of deformation, creep or plastic flow, cold working, elastic afterworking, rupture, shear, and other phenomena hitherto described as anomalous effects but which has been corroborated experimentally in some of its predictions, in particular for the effect of hydrostatic pressure on deformation and compressive strength. The mechanism evolved consists of two processes: (1) an elastic deformation which is a function of the strain or potential energy of the system; failure occurs by "brittle" rupture wherein the maximum extension or maximum internal tension is the criterion; and (2) deformation by means of a two-phase transfer mechanism which is a

function of the thermodynamic potential relations of the system; this type is also a function of time and therefore a function of the rate of application of the load. When both processes of this mechanism are operative, failure occurs by shear; the criterion for this type of failure is given by a function of time, the strain or potential energy, and the thermodynamic potential relations of the system. Expressions are derived for creep or plastic flow of polycrystalline substances from the thermodynamic potential relations which not only satisfy the well-known phenomenon of creep in metals but also express recent empirical creep data of some substances immersed in liquids in which they are somewhat soluble. An expression is also derived for the "brittle" potential type of rupture under combined thrust and hydrostatic pressure. G.R.S.

Quantitative determination of iron by potentiometric titration. D. I. Ryabchikov and V. G. Silnichenko. *Zhur. Priklad. Khim.*, **12**, 1907–11 (1939).—The potentiometric method for iron determination is possible in a great variety of minerals, alloys, nonferrous metals, etc.; it is accurate to within 0.05% and is especially recommended for determining small amounts of iron. The most convenient and simple scheme giving the best results is the oxidation of the investigated solution reduced by a titrated potassium permanganate solution which was previously reduced by a copper chloride solution. P.B. & E.S.

Quantitative spectrochemical analysis of dilute solutions. A safe alternating current high-voltage arc circuit. A. E. Ruehle and E. K. Jaycox. *Ind. Eng. Chem., Anal. Ed.*, **12** [5] 260–61 (1940).—A safe and convenient a.-c. high-voltage arc circuit together with its tie-in with the ordinary d.-c. arc and condensed spark circuits is described. The a.-c. arc is a reproducible source for determining barium, strontium, tellurium, and phosphorus in dilute solutions. A precision of 50 to 100 parts per thousand was obtained using the comparison standard method. Illustrated. See *Ceram. Abs.*, **19** [7] 176 (1940). F.G.H.

Reactions in the solid state (scientific and technical reviews). J. Arvid Hedvall and Günther Cohn. *Kolloid-Z.*, **88** [2] 224–40 (1939).—Reactions in the solid state and related phenomena are discussed under the following headings: (1) definitions; (2) historical summary; (3) thermodynamic relations; (4) types of transitions (active reactions, double transformations, etc.); (5) kinetics of reactions and mechanism, irreversible lattice interference, boundary surface process, interfacial phenomena, quantitative tangible boundary reactions, test methods, and qualitative picture of powder reactions; and (6) possibilities of influencing activity. A few tables give the reactions and products formed at temperatures of 330° to 370°C. of carbonates, sulfates, and silicates: $SrCO_3$, $CaCO_3$, and $MgCO_3$ with $BaCO_3$, $CaSO_4$, $ZnSO_4$, $CuSO_4$, $Sr_3(PO_4)_2$, $Pb_3(PO_4)_2$; and $CaSiO_3$ (wollastonite), $MgSiO_3$ (enstatite), and Al_2SiO_5 (sillimanite). 165 references. L.E.T.

Silicomolybdate method for silica. Harold W. Knudson, C. Juday, and V. W. Meloche. *Ind. Eng. Chem., Anal. Ed.*, **12** [5] 270–73 (1940).—Illustrated. F.G.H.

Synthetic chromium-colored spinel. Joseph Evers. Thesis, Königsberg, 1936, 40 pp.; *Neues Jahrb. Mineral. Geol., Referat. I*, **1937**, p. 199; *Chem. Abs.*, **32**, 4898 (1938).—The material examined consisted of MgO and Al_2O_3 in the (mol.) ratio $1:3^1/_2$, with 0.1 to 15% Cr_2O_3 and two samples containing only 1 mol. Al_2O_3 and 25 to 50% Cr_2O_3. The products were green except for the lower ends of the pear-shaped drops formed, which were rose-red. Those with little Cr were homogeneously green; those with more contained points of red crystal aggregates of ruby. In those with 25% Cr_2O_3 there were alternating zones of red and green. In a few cases of the 50% Cr_2O_3 spinels, metallic Cr appeared with Cr_2O_3 at the base. Analysis showed that in preparation, some Cr is vaporized, the amount increasing with the amount present. MgO vaporized more readily than Al_2O_3. Tempering at 950° caused separation of Al_2O_3 frequently as ruby, this separation being favored by the Cr_2O_3. Up to a certain Cr content, the Cr appeared as red $MgO \cdot Cr_2O_3$, accompanied by a corresponding amount of Al_2O_3 as γ-Al_2O_3, with which, when more Cr was present, part of the Cr formed solid solutions; hardness decreases but density and n increase with increased Cr content. Absorption curves show a maximum at λ 5893 and a minimum at 5016.

Thermodynamics of interfaces in systems of several components. E. A. Guggenheim. *Trans. Faraday Soc.*, **36** [2] 397–412 (1940).—The thermodynamic properties of a plane interface are derived by treating the interface as having a finite thickness. Formulas are derived for the dependence of the interfacial tension on the temperature and the composition of one of the two bounding phases; the pressure, not being an independent variable, is eliminated. The formulas are invariant with respect to the thickness arbitrarily assigned to the interfacial layer. The treatment is correlated with that of Gibbs. The formulas may also be applied to curved interfaces provided that the thickness of the inhomogeneous layer is negligibly small compared with its radius of curvature, a condition usually fulfilled in practice. G.R.S.

Titrimetric method for the determination of gypsum in cement. H. Schaltegger. *Helv. Chim. Acta*, **21**, 180–85 (1938); abstracted in *Referat. Silikatliteratur*, **6** [3] 6259 (1939).—The old classical method of determining SO_4'' as barium sulfate is tedious and long, and S. has succeeded in simplifying and shortening it by determining the SO_4'' as benzidine sulfate. The process is divided into (1) the preparation of the ammonium carbonate extract, and (2) the preparation of benzidine sulfate and the titration of the combined sulfuric acid with 0.025 *N* NaOH. Directions and the necessary reagents and apparatus are given, together with the method of calculation and the preparation of cements for the determination of gypsum. Check analyses which agree well with the sulfuric acid determination after the ordinary barium sulfate precipitation, as well as with the new benzidine precipitation, are tabulated. When the directions are carefully followed, constant values are obtained with the benzidine precipitation and subsequent titration of the combined H_2SO_4 even for small amounts, if the final product is increased by about 0.04% SO_3 as gypsum or in the form of another sulfate. Tests which dissolve out this SO_3 residue are unsuccessful. Even after three extractions with ammonium carbonate the value of 0.04% is not lowered. This result authorized the correction of the final result with 0.04% SO_3. The SO_4'' determination in cement can be made in a half hour with practice, whereas the barium sulfate method requires 3 to 4 times as long. The concentration of the solutions is so selected that the amount of 0.025 *N* NaOH (in cc.) used gives the percentage of SO_3 in the cement. M.V.C.

Viscosity in ternary systems forming open-hearth slags. System FeO–CaO–SiO_2. P. P. Kozakevich, S. P. Leĭba, and E. P. Komar. *Zhur. Fiz. Khim.*, **13**, 248–57 (1939).—A viscosimeter especially constructed for the investigation is described. The viscosities of synthetic slags (system FeO–CaO–SiO_2) at different temperatures were determined. Mixtures which crystallize less readily and form a typical glass upon cooling change their viscosity smoothly but within very large limits. Acid slags with 43 to 48 mol. % of SiO_2 change their viscosity more smoothly. P.B. & E.S.

Volumetric determination of selenate. Glenn C. Soth and John E. Ricci. *Ind. Eng. Chem., Anal. Ed.*, **12** [6] 328–29 (1940).—Illustrated. F.G.H.

PATENTS

Calcium silicate and method of preparing. R. P. Allen (B. F. Goodrich Co.). U. S. 2,204,113, June 11, 1940 (Sept. 30, 1937).—(5) Easily friable precipitated calcium silicate, most of the particles of which have an ultimate size of about 0.3 micron. (7) A process of precipitating finely divided calcium silicate comprises introducing dilute solutions of calcium chloride and of sodium silicate into different portions of a unitary agitated body of aqueous reaction liquid, whereby each reagent is dis-

tributed through a large volume of the liquid before it is brought into reacting contact with the other reagent.

Composition of matter and method of using. I. E. MUSKAT (Pittsburgh Plate Glass Co.). U. S. 2,204,812, June 18, 1940 (Feb. 3, 1938).—A composition of matter suitable for use as a flocculation agent comprises ferric chloride as a major constituent thereof and about 0.5 to 10% of titanium tetrachloride.

Decomposing zirconium ore. LUDWIG TEICHMANN AND HANS MARTINI (I. G. Farbenind. A.-G.). U. S. 2,204,454, June 11, 1940 (Oct. 20, 1938).

Making magnesium silicate products. NORRIS GOODWIN (Hewlings Mumper and Lewis Cruickshank). U. S. 2,203,614, June 4, 1940 (May 6, 1930).

Manufacture of zirconium compounds. DANIEL TYRER. U. S. 2,201,508, May 21, 1940 (April 21, 1939).

Producing alkali-earth silicides. LONZA-WERKE, ELEKTROCHEMISCHE FABRIKEN G. M. B. H. Ger. 682,337, Sept. 28, 1939 (July 17, 1936); IV*b*/12*i*. 38.—Alkali-earth silicides are produced by heating alkali-earth carbonates with silicon carbide in an electric furnace.

D.A.B.

General

Ceramic industry in Ohio. ARTHUR S. WATTS. *Ohio State Univ. Eng. Expt. Sta. News*, **11** [3] 15–20 (1939).—Ceramic raw materials are present in enormous quantity and have high quality and remarkable uniformity. Brick were first made at Marietta in 1788. Stoneware was made in 1799; the Tionesta fire clay is the finest stoneware clay in the country. Yellow glazed tableware was very popular. In 1860 the manufacture of white tableware was started, and East Liverpool is now a world center. The only American porcelain pottery is at Zanesville. Roofing tile was made in 1814. Terra cotta and glazed brick are other products made. Hollow tile manufacture began in 1876. Ohio is the chief producer of wall and floor tile. Drain tile was made in 1832, and Ohio is the leader in quantity and quality. Paving brick is not so important as formerly. Firebrick was first made in 1841, and 300,000,000 brick are now made annually. Silica brick has recently been made from local materials. Art pottery is made in several places. Other important products are sanitary ware, electrical insulators, chemical stoneware, grinding wheels, etc. W.D.F.

Ceramics in the Division of Geology and Geography, National Research Council. ROBERT B. SOSMAN. *Bull. Amer. Ceram. Soc.*, **19** [6] 221 (1940); see *ibid.*, **18** [2] 76–77 (1939).

Composition of stone, etc., in relation to silicosis: I and II. A. SHAW. *Proc. S. Wales Inst. Engrs.*, **55**, 96–142 (1939); see *Ceram. Abs.*, **18** [8] 224 (1939). L.R.B.

Determination of dust concentrations in mine atmospheres. J. HOWARD GRIFFITHS AND T. DAVID JONES. *Colliery Guardian*, **160**, 671–72 (1940).—Instead of a piece of filter paper to catch the dust, a Soxhlet extraction thimble is used. It is mounted on a glass tube which is put in a rubber stopper; this fits in a copper holder on the sampler. The sampler holds three of these tubes, so that three samples may be taken before reloading. Air flow is measured by a dry meter, and a pump supplies the suction; the open end of the tube is pointed into the air stream. Tests showed that the best sampling velocity is the same as that of the air stream. If the sampling velocity is less than this, coarse particles in air deflected outside of the tube will tend to continue into the tube by their own momentum, and the sample will have too many coarse particles; if the sampling velocity is greater, air will be deflected from the outside into the tube, and coarse particles will tend to go outside the tube. W.D.F.

Developments in ceramic engineering during the past ten years. ARTHUR S. WATTS. *Ohio State Univ. Eng. Expt. Sta. News*, **12** [1] 19–20 (1940).—The developments include (1) elimination of associated minerals from American clays by electroosmosis, (2) beneficiation of American clays by flotation combined with the use of electrolytes, (3) introduction of nepheline syenite in glass, (4) introduction of pyrophyllite in commercial ceramic bodies, (5) introduction of talc in commercial dinnerware to resist heat shock, (6) use of the casting process for large, massive products and refractories, (7) use of the semidry mixing process for many fine ceramic bodies, (8) the de-airing pug mill to remove entrapped air, (9) use of humidity control in drying, (10) rapid firing of ceramic ware, (11) automatic temperature and atmosphere control for ceramic kilns, and (12) controlled cooling of tunnel kilns for annealing. W.D.F.

Diet of workers in porcelain industry. W. SCHMIDT-LANGE AND E. REICHEL. *Arch. Hyg. & Bakt.*, **122** [2–3] 138–58 (1939).—Net findings are expressed in a composite average worker of the three selected persons, who weighed 70 kgm. and who consumed, during the 10-day period, 117.4 gm. proteins (55% of which was of animal origin), 164 gm. fats, and 347.7 gm. carbohydrates. Compared with food values approximated earlier in Bavaria these heightened intakes represent what is apparently termed "Americanization" and also "citification" of this industrial population. The authors hint that some lowering here and there might occur without actual detriment to working capacity. K.R.

Dust in the foundry; its consequences and prevention with regard to silicosis. U. GABINO. *Ind. Meccan.*, **22** [2] 86–95 (1940).—The problem of dust in the foundry, its prevention, the equipment used for precipitating and exhausting the dust at its place of origin, the regulations by state health boards (particularly those in force in the U. S.), and the clinical aspect are discussed at length. The recommended preventive measures include (1) avoiding as much as possible the employment of siliceous materials in dust-producing processes, (2) reducing to a minimum the dust in suspension in the air near the workplaces, (3) prohibiting this perilous work for men physically not fit for it, and (4) periodic visits by physicians and eventually the taking of radiograms to detect the start of the disease. The quantity of dust in the air should be reduced (1) by precipitators or exhausters as close as possible to the place of origin and (2) by isolating the dust-producing workplace; the operators should be protected by efficient gas masks. 28 references. M.H.

Empire services of the Imperial Institute in wartime. HARRY LINDSAY. *Jour. Roy. Soc. Arts*, **88** [4560] 550–68 (1940).—The Imperial Institute was founded in 1887 as a memorial of Queen Victoria's golden jubilee. Its present constitution (1925) comprehensively includes the responsibilities and ideals of the Institute in promoting the commercial, industrial, and educational interests of the empire. L. shows how these aims are being carried out in wartime. The laboratories, scientific and technical, are numerous; their work is sketched in considerable detail. A special laboratory is well equipped for ceramic work. Practical small-scale trials can be made to determine whether clays are suitable for making brick, tile, porcelain, stoneware, china, earthenware, or refractory products. The apparatus available includes a clay-washing plant, a brick or tile press, an auger machine, a potter's wheel, etc., and the furnaces necessary for firing test pieces at the appropriate temperatures. One furnace is capable of reaching a temperature of 1750°C. Another laboratory is fitted for testing raw materials to determine their suitability for use in making natural or Portland cement, hydraulic lime, and natural or artificial pozzuolana. Portland cement can be made on a small scale in a specially designed furnace, and the product can be tested according to standard specification requirements. Manufactured cement can, if required, be tested for accord with British or other specifications. Mineralogical

investigations are of considerable importance and frequently accompany the chemical work. The department is equipped for complete mineralogical examinations by microscopic and other processes used in determining rocks, minerals, ores, and concentrates of all types. It is frequently possible from the results obtained to offer opinions upon the commercial value of a mineral product or to give advice on methods of preparation for the market. Chemical and mechanical analyses can also be made of soils. K.R.

Fatal subacute occupational lead poisoning. WERNER EHRHARDT. *Arch. Gewerbepath. & Gewerbehyg.*, **9**, 407–13 (1939).—Industrial lead poisoning as a rule is a chronic poisoning. Characteristic symptoms appearing suddenly, like the colic and intestinal stoppage, show that the poison has been at work in the system for a long time. Acute poisoning as an industrial disease is seldom encountered. E. reports an acute case which came to the Berlin Clinic for Industrial Diseases and discusses its manifestations. On Dec. 4, 1938, a sprayer of lead, 49 years old, was brought to the clinic in a dangerously ill condition. Very carefully and fully he was examined; the next day he died. For several weeks as an occasional worker he had been spraying cable muffles in a cable plant. The workspace was 3 x 4 x 3m.; the spraying was expected to be done under a hood. The worker used a spray pistol in which a lead wire was inserted and the propane gas flame mixed with air, which was introduced into the pistol at the same time, melted the lead wire. The molten lead was at once ejected by the pressure of the air, spraying the muffles revolving in front of it. During this process the worker was expected to wear a mask and to manipulate the pistol through slits. About the middle of November he had been seized with violent pain and vomiting; he was off from work for 8 days and resumed work again late in November. The hood apparatus was then out of repair; three other men working in his absence in the same small space, with very low suction in the ventilating apparatus, caused the returned worker to ask for the night shift where he could be alone. On November 30 he was overcome with the same violent symptoms as before. Being somewhat better by the next night, he insisted on working, only to return home in the same condition, with additional symptoms; his wife noted that his face was as black as lead. E. details the man's further breakdown and the efforts of the company physician in palliation; these failing, the victim was transferred on Dec. 4 to the Occupational Clinic. The plant conditions under which the lead spraying took place were examined and reported on by E. after the autopsy. A large hole was found in the left side of the framework supporting the hood through which the sprayed lead had been escaping into the small enclosure; lead dust lay heavy on the glass valves of appliances carrying the cables; the clothing and shoes worn by the various shifts were loaded and sodden with the scattering lead dust; the mask of the worker was in disrepair, so that he might not have worn it on his final night shifts. He must have breathed the lead dust in its most finely divided particles, i.e., in almost molecular form. Such an aerocolloid condition is taken up by the organism with special ease and constitutes the highest possible risk to the subject. The lungs in this case were permeated with it, and the blood readily carried this fatal dust to the entire system. From the evidence derived from the illness, the autopsy, and the plant, E. concludes that the poisoning could be called acute but also subacute, the worker evidently having been sensitized by his first attack and the subsequent exposures, massive in character, sustained December 1 and 2. An inspector testified that, after being placed on the night shift, this worker had done the work of three ordinary men on the spraying. Hence his constitutional resistance was utterly broken down beyond repair. The lead content of the urine was 2140 γ in 10 gm. of dry feces, 490 γ in 10 gm. of the liver, and 370 γ in 10 gm. of the kidneys. K.R.

Fuel gas generator. HARRY A. CURTIS, RALPH B. STITZER, AND WILBER J. DARBY. *Ind. Eng. Chem.*, **32** [6] 757–62 (1940).—Developments in the use of electrical energy as a source of heat for the carbonization of coal are described. Illustrated. F.G.H.

Glass industry of Ohio. ARTHUR S. WATTS. *Ohio State Univ. Eng. Expt. Sta. News*, **11** [4] 10–13 (1939).—Ohio is the third largest glass-producing state and has twenty glass plants. In 1797 there was a glass plant on the Ohio river; by 1825 they were all over the state. The production of hollow glassware has increased twenty times since glass blowing machinery has been introduced. Ohio is an important producer of flat glass. Today this is drawn from the tank as a continuous flat sheet. Other important products are electric light bulbs, fiber glass, and glass blocks for use in building construction. W.D.F.

Plant laboratory. K. SPINGLER. *Tonind.-Ztg.*, **62** [44] 492–93 (1938).—The plant laboratory has the following tasks: the testing of raw materials and half-finished and finished products, the working out of new formulas, improvement of old shapes and construction of new shapes, and local research. W.K.

Prevention of industrial dermatitis. J. V. KLAUDER, E. R. GROSS, AND H. BROWN. *Arch. Dermatol. & Syphilol.*, **41**, 331 (Feb., 1940).—Much of the industrial skin disease is caused by the methods of cleaning the skin. Skin cleansing, methods and materials used, and the use of protective applications are studied. Formulas for eight protective applications are given. Substitutes for mechanic abrasive soap are listed. Various kinds of soaps and detergents, such as triethanolamine soap, naphthenic acid soap, and sulfonated oils, are discussed. The detergent properties of the vegetable meals are not sufficiently appreciated. F.S.M.

Pyrophyllite dust; its effect and control. M. F. TRICE. *Amer. Inst. Mining Met. Engrs. Tech. Pub.*, No. 1179; *Mining Tech.*, **4** [3] 13 pp. (1940).—North Carolina is the only commercial source of pyrophyllite at present. Pyrophyllite occurs associated with quartz, together with chloritoid and sericite. All the pyrophyllite marketed is ground; it is classified by air separation. It is mined by open-cut, glory-hole, and underground methods. At only one operation has work been carried on since 1920. Underground, the free silica content of the dust is 35%; 80% of the particles are less than 3 microns in size; dust concentration rises to 500 million particles per cu. ft. when drilling. X-ray examination was made of 101 workers; of those with over two years' exposure, 35% were affected; two died. The lesions were the same as those produced by free silica alone. The threshold dust concentration allowable underground is 10 million particles per cu. ft. Drilling is done wet; stopes are ventilated by "ventubes." Above ground, tight housing for machinery and exhaust ventilation have been installed. These procedures give the required results. W.D.F.

Recent Commission findings. ANON. *Ohio Industrial Commission Monitor*, **13**, 41–42 (March, 1940).—An employee of a steel foundry worked from May, 1922, to June, 1939, in the cleaning department in the foundry, where his duties required him to remove hardened sand from castings by use of a pneumatic hammer. A dust count in the room showed an exposure well below the accepted safe standard, but the dust contained approximately 20% of free silica. The Claims Referee was of the opinion that the exposure to silica dust was well established, and the claim was approved as compensable by the Silicosis Referees. Acting under the provisions of an amendment to the Occupational Disease Act effective May 26, 1939, the Commission granted an award of $6500 to the widow and minor children, payable at the maximum rate of $18.75 per week. F.S.M.

Recent developments in relation to silicosis. LEROY U. GARDNER. *Ind. Med.*, **9**, 45–51 (Feb., 1940).—Certain tentative conclusions derived from experimental work upon silicosis in progress at the Saranac Laboratory for some years are recorded. As a basic doctrine, G. considers that it is still true that only certain forms of free silica and the group known as asbestos (fibrous silicates)

are capable of producing fibrosis in a normal lung. The trend of some of the Saranac experiments, however, has altered certain concepts previously regarded among research workers as fixed. Inhaled chrysotile asbestos fibers now appear to be irritating, not because they are silicates, but because they are stiff fibers, mechanically irritating to the lungs. Grinding them down very finely so that few of the fibers were longer than 2 microns practically destroyed this irritating property. Even with an average atmospheric concentration of 125 million particles per cu. ft. of air, no fibrosis developed. If the effect of asbestos on the lung were entirely chemical, a decrease in particle size might be expected to increase tissue response. Large particles of free silica have little effect on the body, but small particles increase the reaction of body cells. The histology of early asbestosis, as detailed by G., does not suggest chemical injury. Among dozens of different silicate minerals, only the group of five known as asbestos are unique in having fibrous structure and in being commonly recognized as pulmonary irritants. The group varies in chemical composition within itself more markedly than, as a group, from other silicates. Chrysotile asbestos has the same chemical formula as a nonfibrous silicate, serpentine, which is physiologically inert. In mills that fabricate asbestos textiles there may be much fine dust, but if the dust is composed of serpentine and very short chrysotile fibers, clinical asbestosis will not develop. Free silica dusts, unlike those from asbestos, cause a chemical form of irritation in laboratory animals. G. describes the two histological stages which characterize the tissue reaction in such animals after inhaling excessive quantities of pure quartz or flint dusts in an extremely fine state. In human subjects, only cases of rapidly developing silicosis show evidence of this two-stage reaction. The solubility hypothesis, however, hitherto rather widely used in interpreting silica reactions in the lungs, is declared to rest entirely on indirect evidence. It has never been possible to prove that silica dissolved within the body, because the soluble material immediately combines with local tissue elements. Soluble silica detected in the urine and the blood is now generally accepted as more likely derived from food and drink than from inhaled dust. G. describes many of the Saranac experiments whose outcome individually and collectively is difficult to reconcile with the theory of silica solubility. No fibrosis has ever been produced by single or repeated injections of soluble silica in its various forms. Evidences of specific toxicity exerted by silica upon living tissues have been manifested through long series of experiments lasting years; many concrete examples are given. Intravenous experiments immediately fatal to guinea pigs produced no symptoms in dogs. The toxic effects of quartz have been more or less neutralized by adding small amounts of different substances, e.g., colloidal aluminum hydroxide which gives complete protection in a 0.03% dilution. Particle size is discussed as having new and unexpected relations with both highly toxic and merely slow fibrotic conditions. G. is cautious about drawing final conclusions from animal experiments alone, but he does begin to correlate his experimentally derived facts into a coherent silicosis etiology. K.R.

Relation between the thermal expansion and the properties of ceramic products. W. H. EARHART. *Ohio State Univ. Eng. Expt. Sta. News*, **12** [2] 25–28 (1940).—The composition of a body may be modified to obtain a desired thermal expansion. The thermal history also affects the thermal expansion; certain compositions fired at low temperatures have less expansion than the same body fired at higher temperatures. Thermal expansion may be measured directly on a bar or by the interferometric method. Bodies with a considerable amount of high-expansion quartz in low-expansion clay may show dunting tendencies on cooling. Beta-quartz expands on cooling and may also set up strains. The abrupt quartz inversion point at 575°C. may be destructive. Quartz and clays have fairly uniform thermal expansions, but the feldspars are variable. Hotel china, sanitary ware, and floor tile all have thermal expansion curves lying close together. The quartz present does not seem to affect them. A quartz grain shrinks more rapidly on cooling, and it is thought to pull away from the remainder, so that it does not fill its recess in the body completely on cooling. W.D.F.

Report of Committee on Ceramic Education. N. W. TAYLOR, *Chairman*. *Bull. Amer. Ceram. Soc.*, **19** [6] 220–21 (1940).

Report of Committee on Publications. JOHN D. SULLIVAN, *Chairman*. *Bull. Amer. Ceram. Soc.*, **19** [6] 222–23 (1940).

Report of Committee on Research. ARTHUR A. WELLS, *Chairman*. *Bull. Amer. Ceram. Soc.*, **19** [6] 214–18 (1940).

Report of Committee on Sections and Divisions. M. E. HOLMES, *Chairman*. *Bull. Amer. Ceram. Soc.*, **19** [6] 221–22 (1940).

Report of Committee on Standards. JOHN W. WHITTEMORE, *Chairman*. *Bull. Amer. Ceram. Soc.*, **19** [6] 218–20 (1940).

Roentgenologic aspects of bronchomycosis. H. P. DOUB. *Radiology*, **34**, 267–75 (March, 1940).—The signs and symptoms of the bronchomycoses are similar to those of tuberculosis and they are often at first thought to be tuberculous in origin. D. describes some of the more prominent Roentgen signs of the various bronchomycoses. F.S.M.

Silicosis. E. R. HAYHURST. *Merck Rept.*, **49**, 6, 8, 9, 28, 30, 32 (April, 1940).—H. gives an excellent short review of silicosis. F.S.M.

BOOK

Engineers and Engineering in the Renaissance. WILLIAM BARCLAY PARSONS. Williams & Wilkins Co., Baltimore. 661 pp. Price $8. Reviewed in *Science News Letter*, **37** [12] 192 (1940).—P. discusses the amazing engineering achievements produced during the Renaissance. P.G.H.

PATENT

Coating and coloration of granulated materials. N. P. HARSHBERGER (Bakelite Building Products Co., Inc.). U. S. 2,202,002, May 28, 1940 (June 2, 1936).—A roofing granule has on the exterior thereof a coating comprising a hardened high-alumina cement containing principally calcium aluminates or calcium alumina ferrites and substantially free of free lime in amount to produce objectionable efflorescence in exposure, the surface of the granule being also substantially resistant to chipping and wear and stable with respect to a bituminous bond.

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